



NATO Advanced Research Workshop
Metallic Materials with High
Structural Efficiency

High Strength Microstructural Forms in Titanium Alloys Processed with Rapid Heat Treatment

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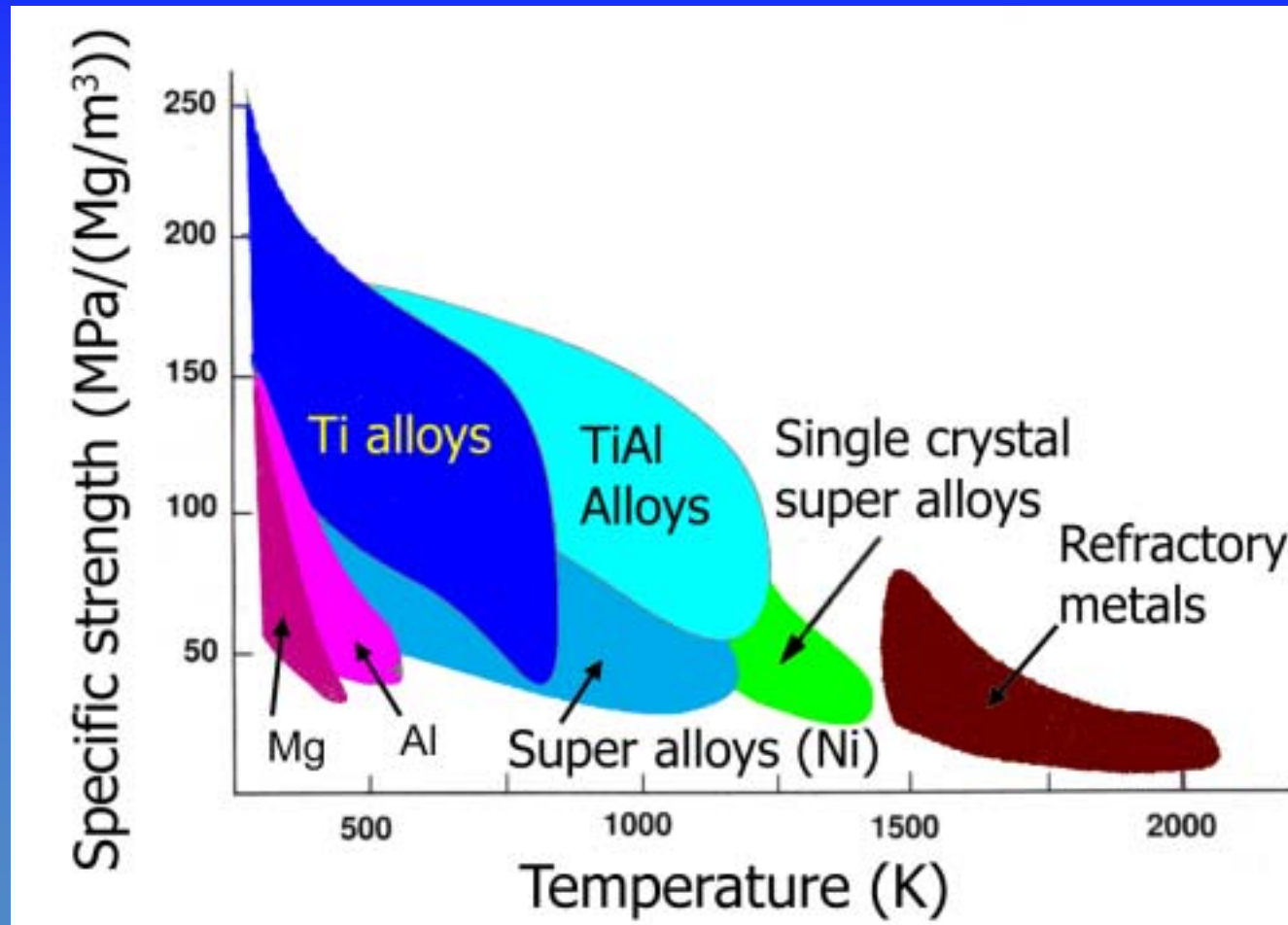
Outline

- Introduction:
 - Strengthening of titanium alloys
- Background of rapid heat treatment (RHT)
 - microchemical inhomogeneity
 - grain growth
- Examples of RHT:
 - alpha/beta alloys
 - beta alloys
- Super strength beta alloys
- Modulated structure of alpha double-prime martensite
- Texture Controlled Grain Growth Kinetics at Continuous Heating
- Conclusions



Specific Strength of Constructional Materials

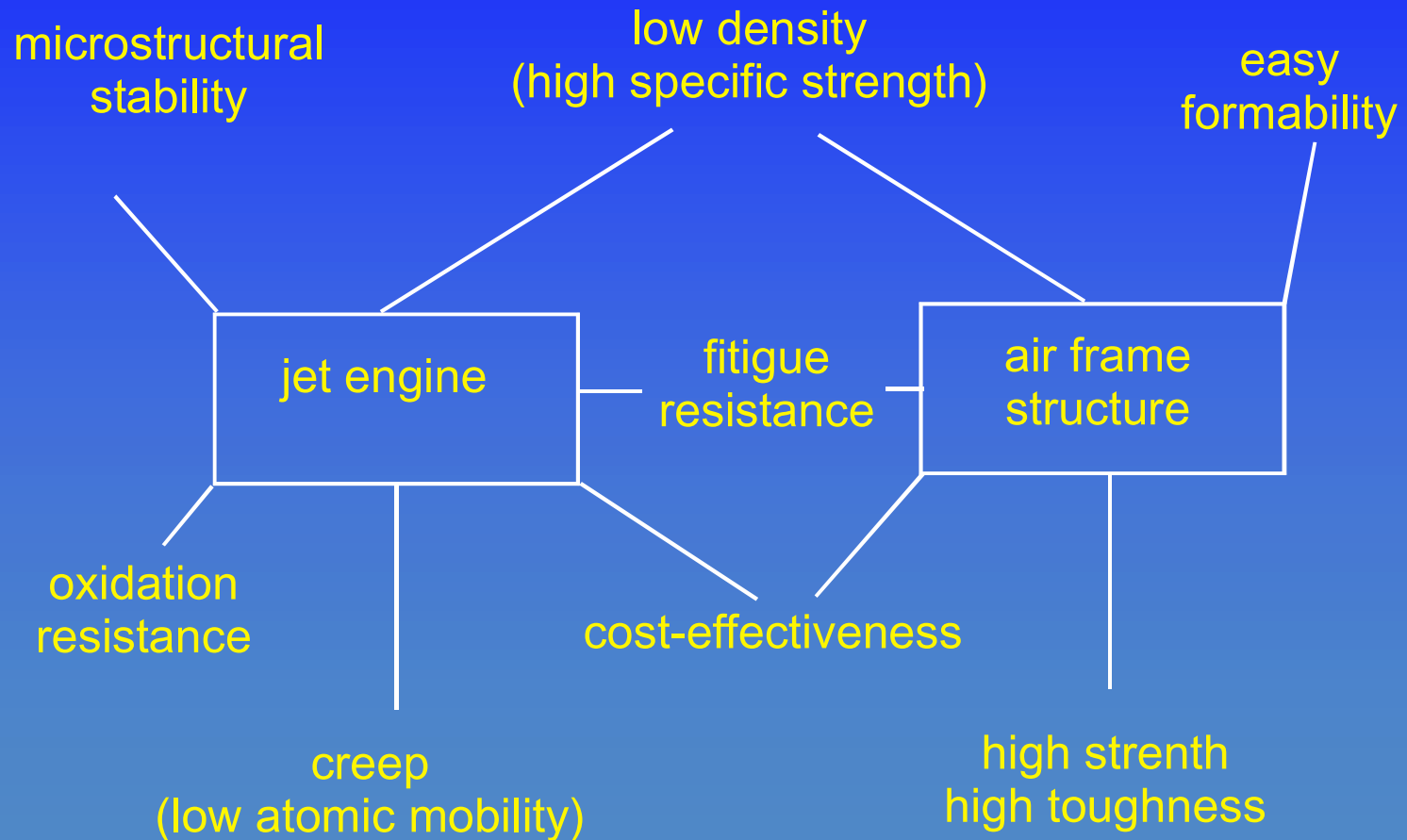
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Property Requirements for Titanium Alloys in Aircraft Applications

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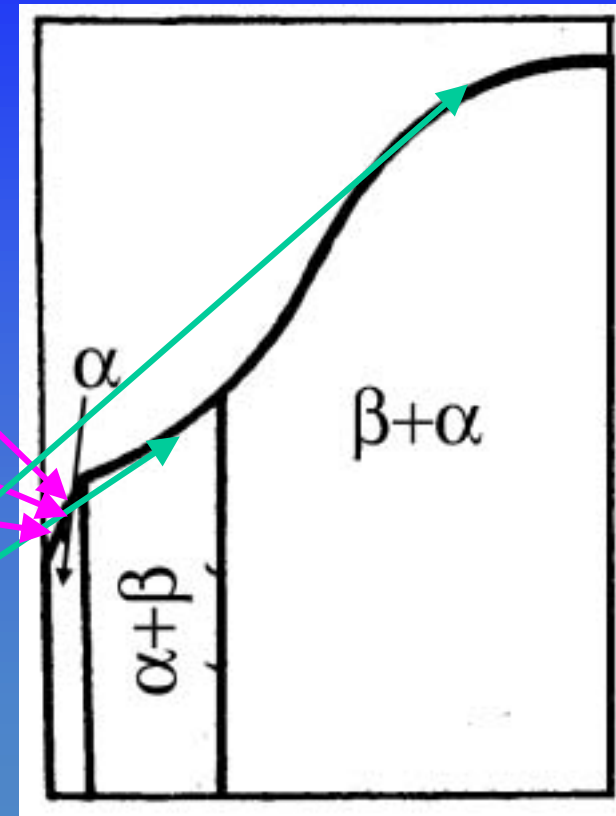




Strengthening Mechanisms of Titanium Alloys

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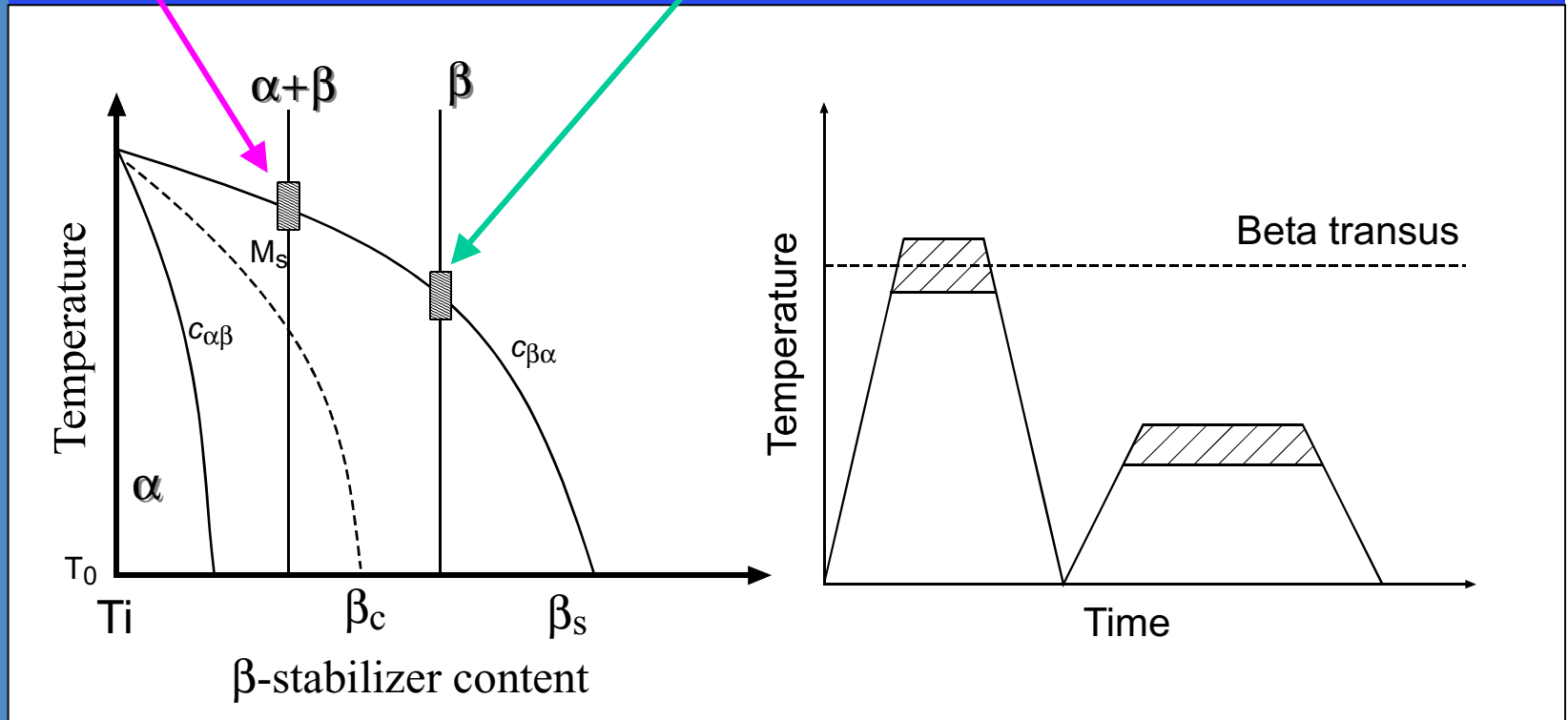
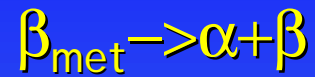
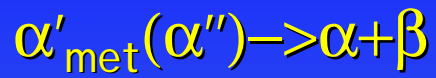
1. Solid solutional (alloying of α -solid solution)
2. Textural
3. Dispersional
4. Heterogeneous (α (h.c.p.) $\leftrightarrow\beta$ (b.c.c.) phase transformation)





Heat Treatment

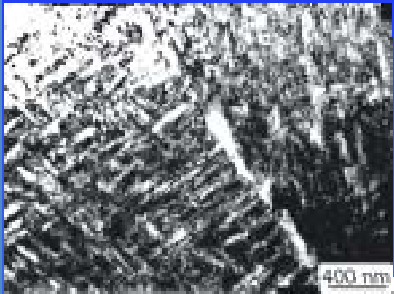
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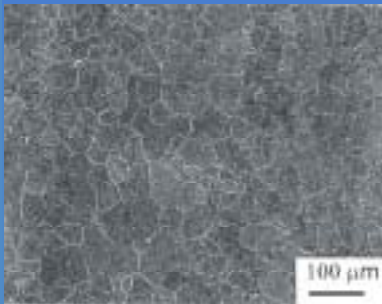
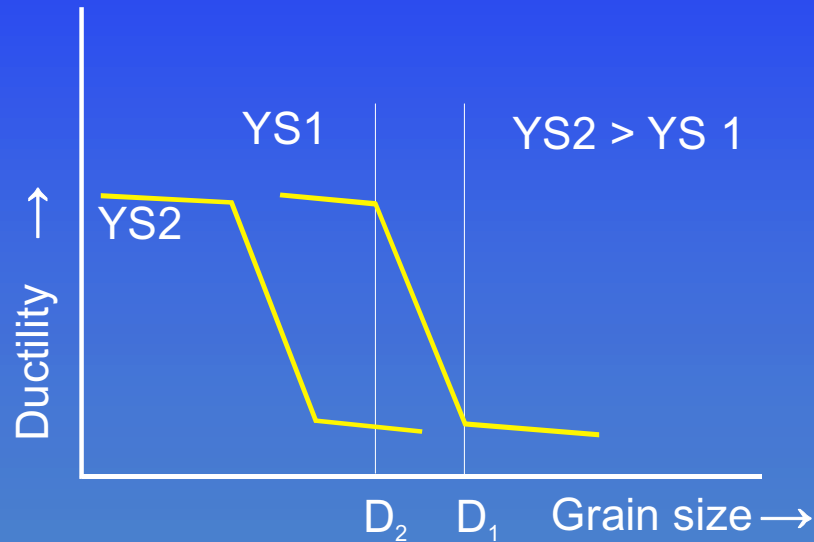


Grain Size Dependence of Ductility (scheme)

YS1

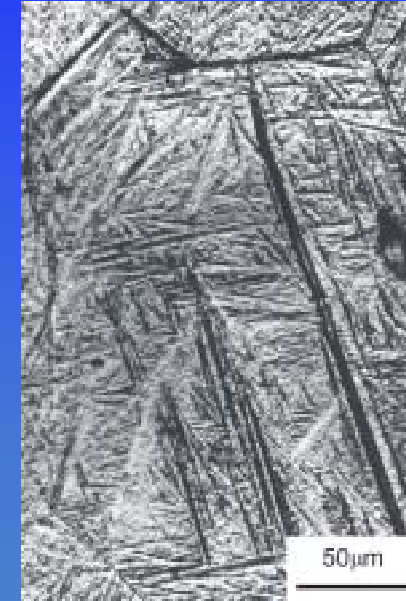
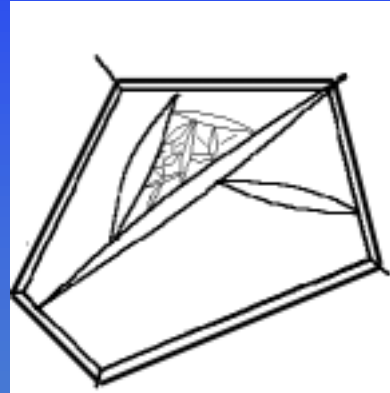
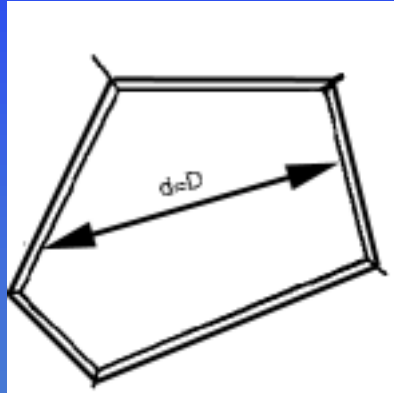


YS2



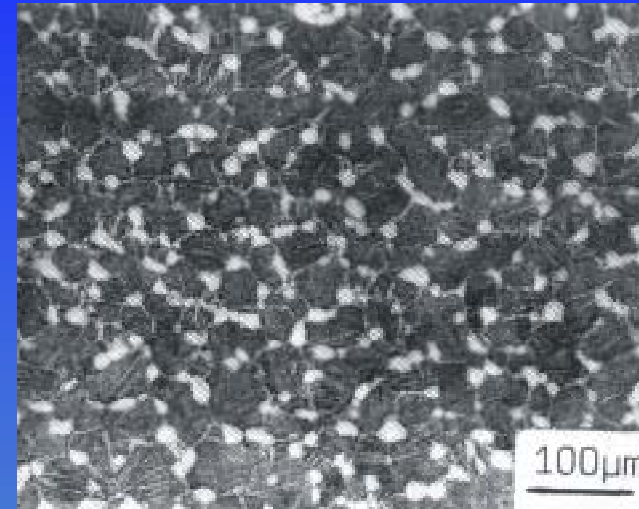
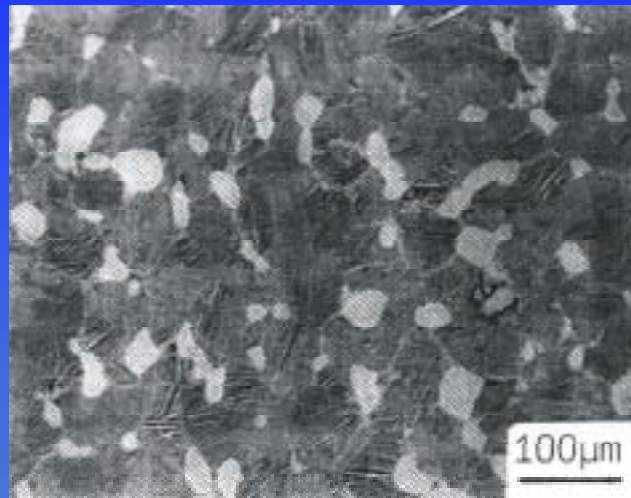


Conventional Heat Treatment





Compromise: Bimodal Microstructures



- sensitive to α_{pr} volume fraction and size due to:
partitioning effect;
 α_{pr} texture.
- therefore needs very careful processing



Background of Rapid Heat Treatment (RHT)



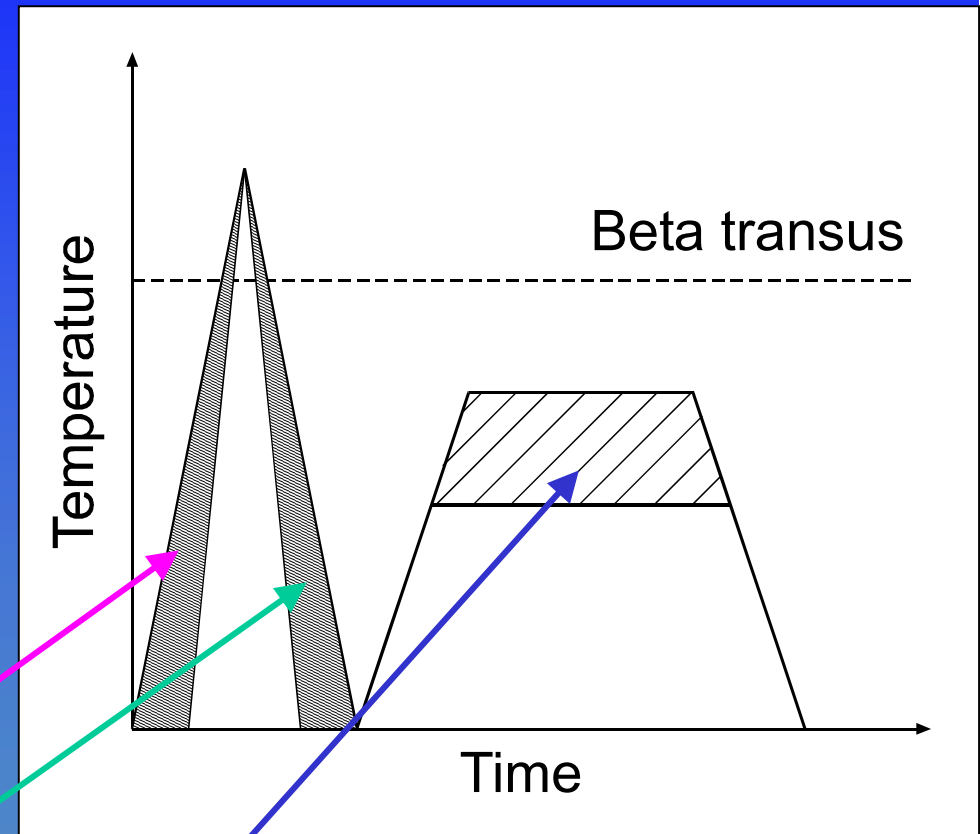
RHT Background

TECHNICAL ESSENCE:

- rapid continuous heating above β -transus followed by instantaneous cooling;

Controlled parameters:

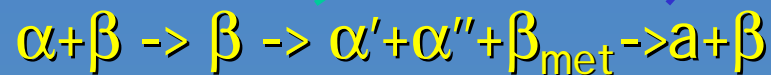
- heating rate;
- peak temperature;
- cooling rate;



rapid
heating

cooling

aging

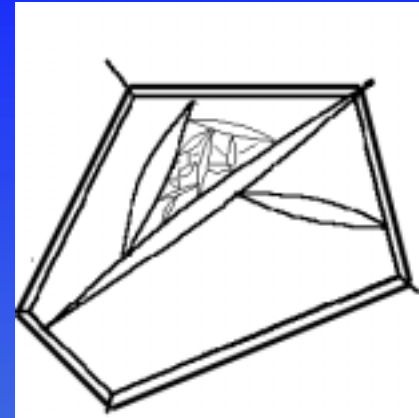




RHT Background

MICROSTRUCTURAL FEATURES:

- full dissolution of primary α ;
- small beta-grain sizes;
- fine lamellar/acicular intragrain microstructure formed through:
 - in $\alpha+\beta$ alloys:
 - martensite type transformation
 - diffusion controlled transformation
 - in β alloys:
 - precipitation hardening



Conventional heat treatment

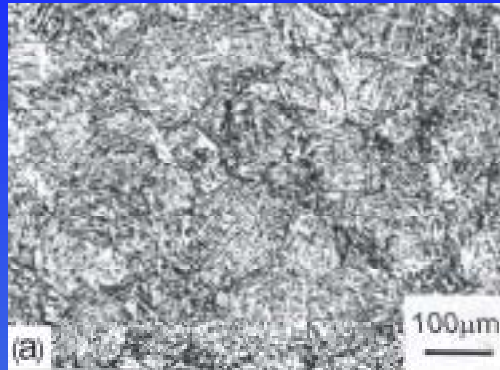


RHT

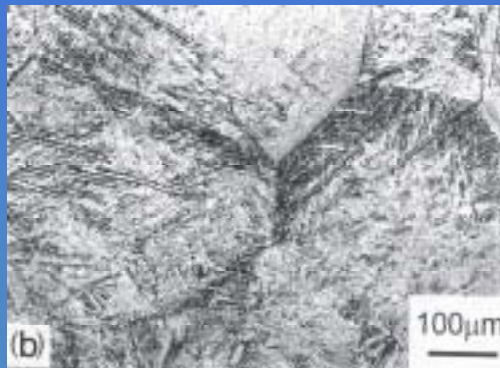


Fine-Grained and Coarse-Grained Microstructures

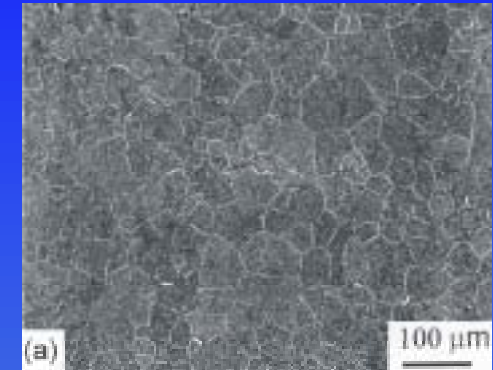
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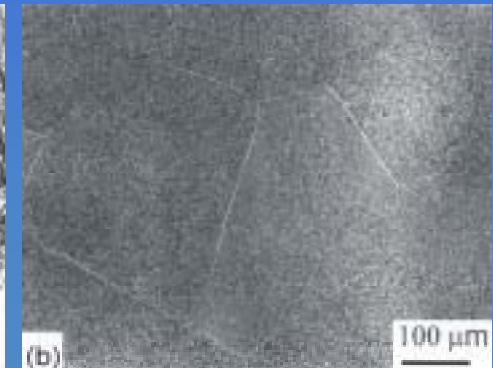
VT18Y



VT6



β-CEZ





Grain Structure Evolution on Heating

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Isothermal conditions:

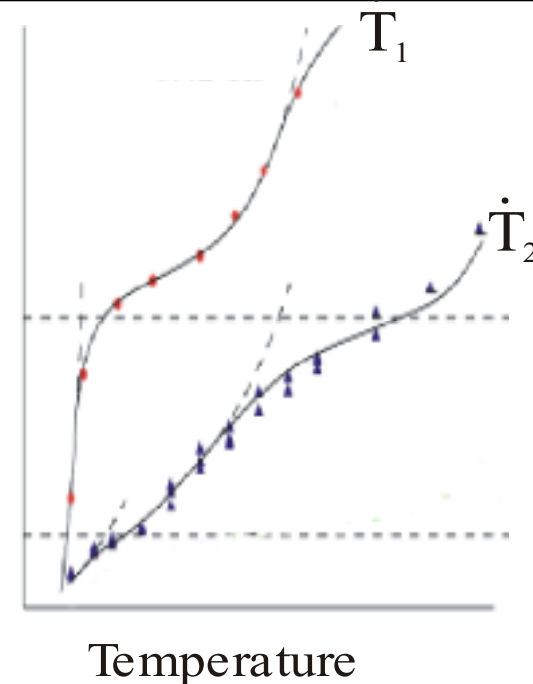
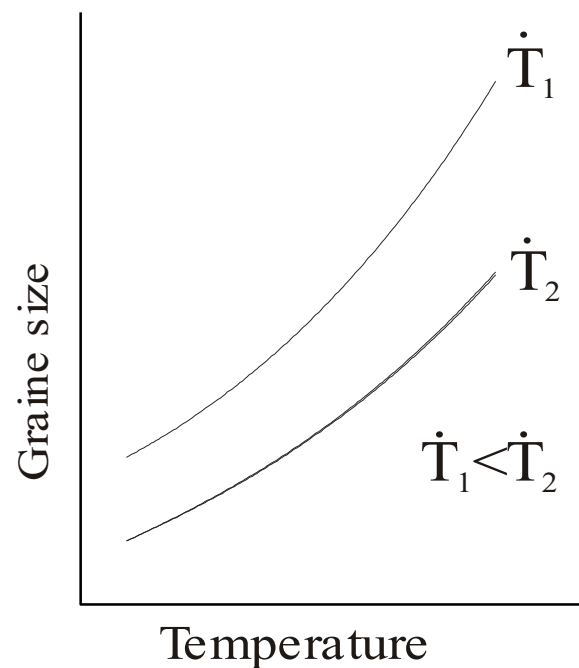
$$D^n - D_0^n = Kt \exp(-Q/RT)$$

Continuous heating

$$D^n - D_0^n = (KR/\dot{T}Q) \left\{ \left[T_f^2 \exp(-Q/RT_f) \right] - \left[T_i^2 \exp(-Q/RT_i) \right] \right\}$$

Isotropic condition

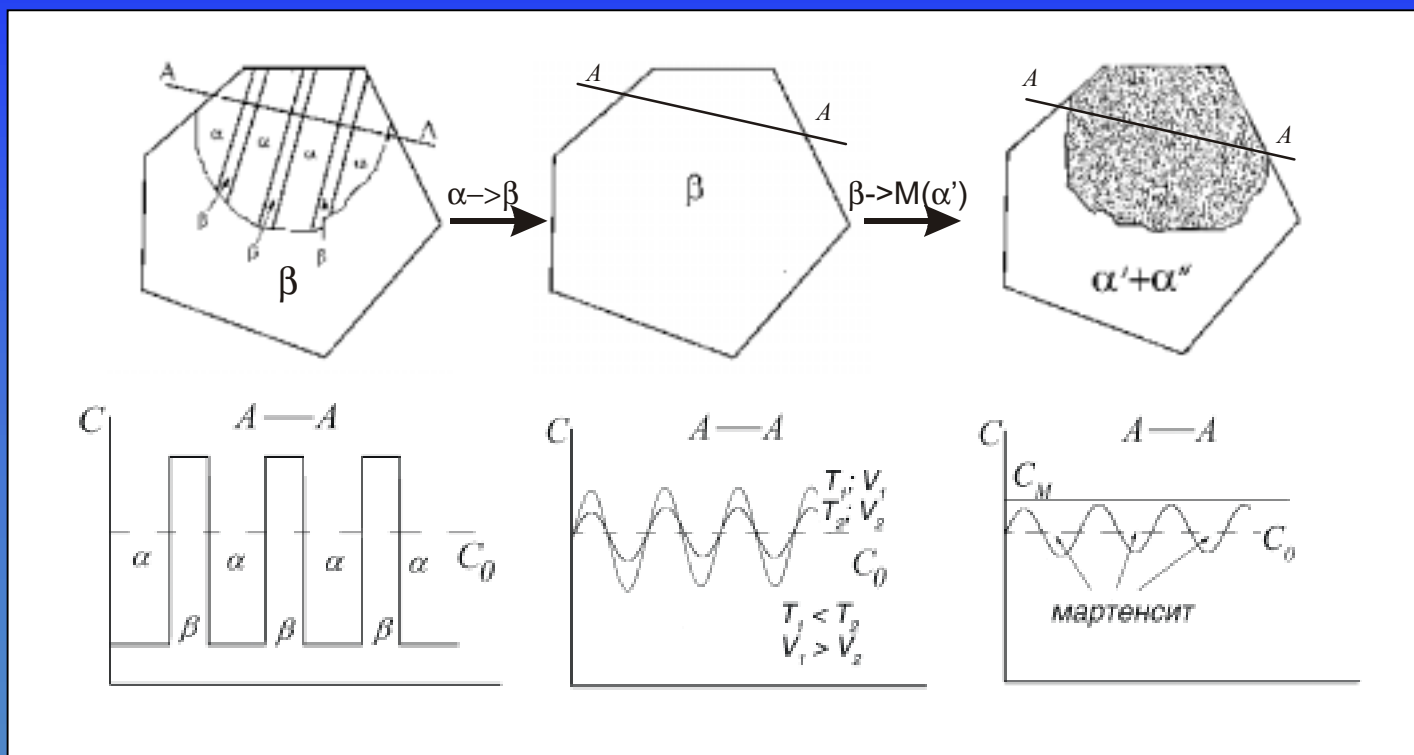
Textured condition





Microchemical Inhomogeneity

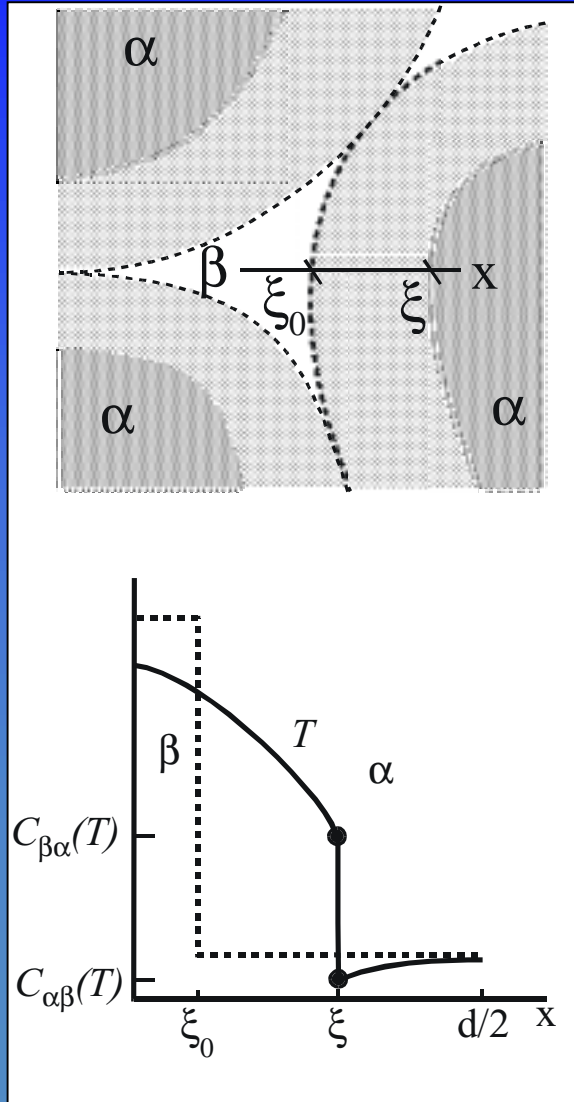
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Microchemical Inhomogeneity

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$$(C_{\beta\alpha} - C_{\alpha\beta}) \frac{d\xi}{dT} \dot{T} = -D_{\alpha}(T) \left. \frac{\partial C_{\alpha}}{\partial x} \right|_{x=\xi^{+}} + D_{\beta}(T) \left. \frac{\partial C_{\beta}}{\partial x} \right|_{x=\xi^{-}}$$

$$\frac{\partial C_{\beta}}{\partial T} = \frac{1}{\dot{T}} D_{\beta}(T) \frac{\partial^2 C_{\beta}}{\partial x^2}; \quad 0 \leq x \leq \xi$$

$$\frac{\partial C_{\alpha}}{\partial T} = \frac{1}{\dot{T}} D_{\alpha}(T) \frac{\partial^2 C_{\alpha}}{\partial x^2}; \quad \xi \leq x \leq \frac{d}{2}$$

Starting conditions:

$$C_{\alpha}(x, T_0) = C_{\alpha_0}; \quad C_{\beta}(x, T_0) = C_{\beta_0}$$

Boundary conditions:

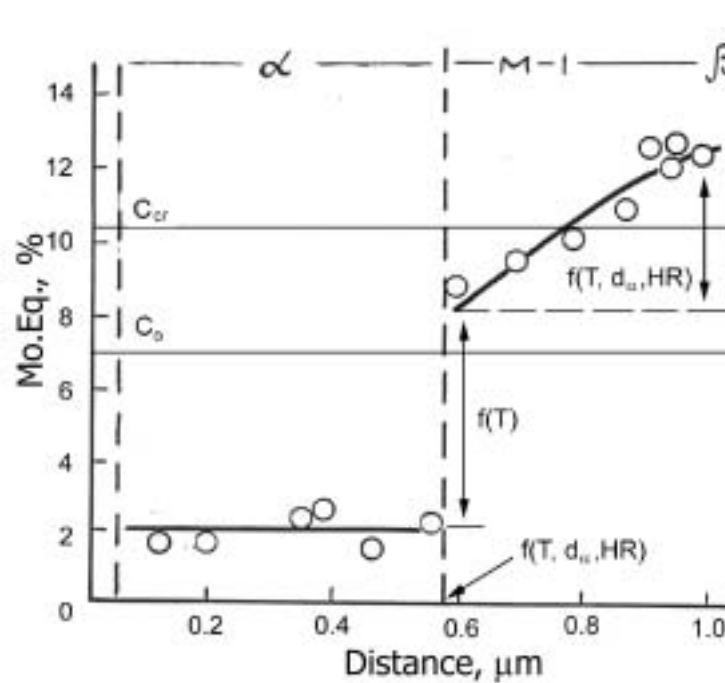
$$C_{\alpha}(\xi, T) = C_{\alpha\beta}(T); \quad C_{\beta}(\xi, T) = C_{\beta\alpha}(T)$$

$$\left. \frac{\partial C_{\alpha}}{\partial x} \right|_{x=d/2} = \left. \frac{\partial C_{\beta}}{\partial x} \right|_{x=0} = 0$$

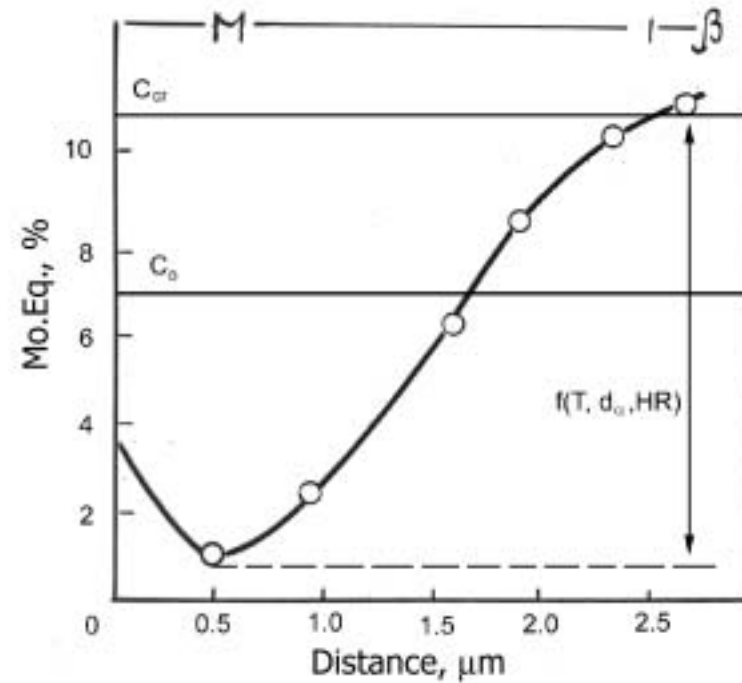


Microchemical Inhomogeneity in VT23 Alloy (STEM)

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$\alpha+\beta$ ST



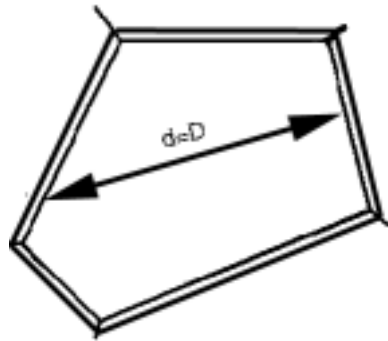
β ST



Strength

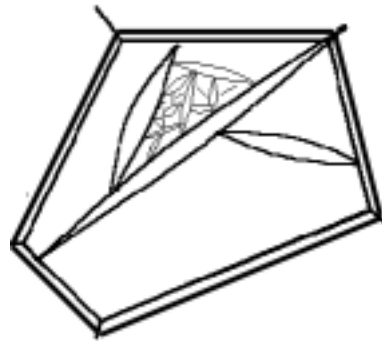
$$\sigma = \sigma_0 + kd^{-1/2}$$

Unfragmented grain



$d=D \approx 100 \mu\text{m}$

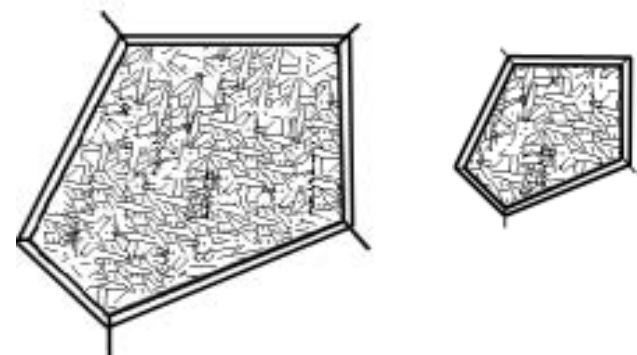
Fragmentation in
homogeneous phase



$d \approx 1-10 \mu\text{m}$

$D \approx 100 \mu\text{m}$

Fragmentation using volume
concentrational modulation



$d \approx 0.05-0.5 \mu\text{m}$

$D \approx 100 \mu\text{m}$

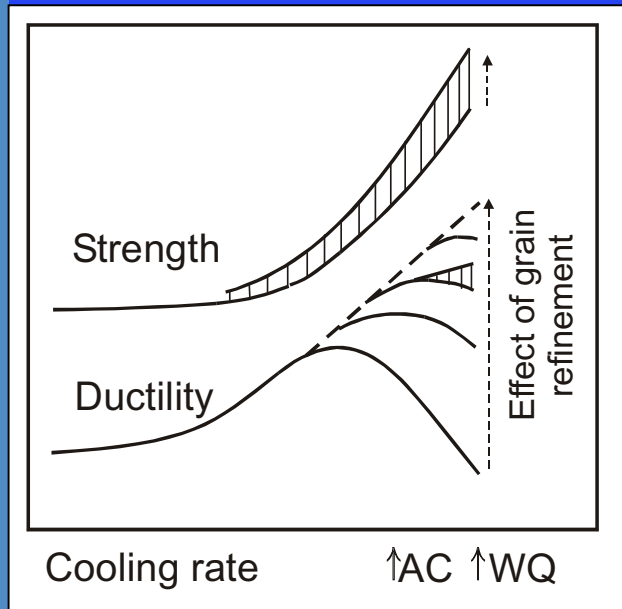
$D \approx 10 \mu\text{m}$



Examples of RHT



Strength/Ductility Relationship in $\alpha+\beta$ Alloys



Ti-6Al-4V; WQ

grain size 500 μm : YS = 1105 MPa;

A5 = 4.6%;

grain size 80 μm : YS = 1105 MPa;

A5 = 9.9%;

grain size 30-50 μm : YS = 1268 MPa;

UTS = 1349 MPa;

A5 = 10.9%

VT23 (Ti-0.5Al-2.0Mo-4.5V-1Cr-1Fe); WQ

grain size 300 μm : UTS = 1350 MPa;

A5 = 1%;

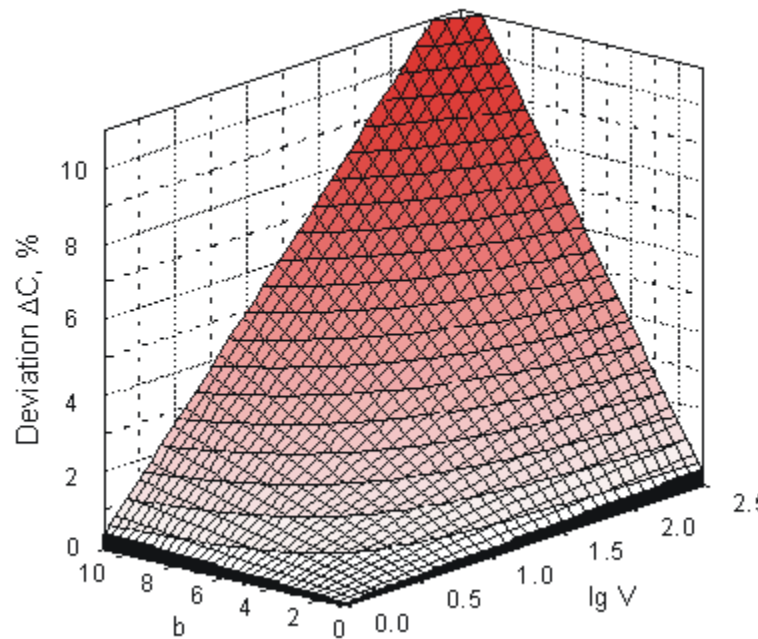
grain size 20 μm : UTS = 1900 MPa;

A5 = 6%;



Microchemical Inhomogeneity of β -Phase

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Ti-6Al-4V; $C_0=2.66$

$$\Delta C = f(C_0, d, \dot{T}, T_p)$$

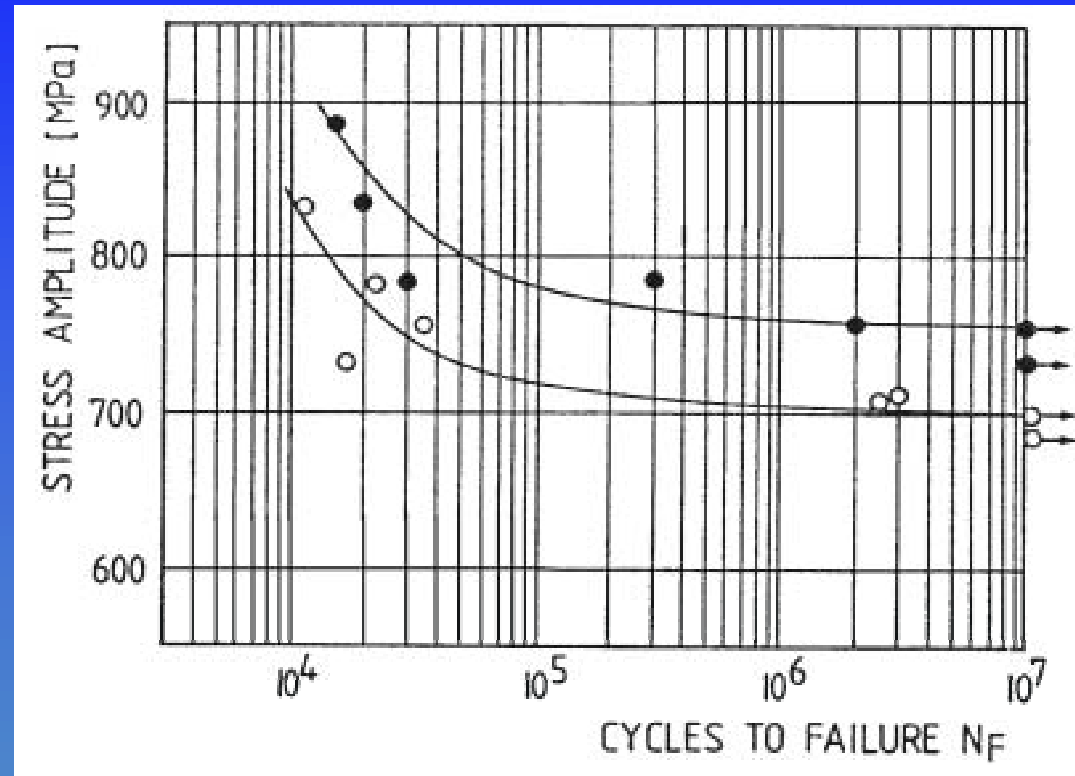
For $C_0 = \text{const}$; $T = T_\beta$

$$\Delta C = f(d, \dot{T})$$



S-N Curves of Ti-6Al-4V

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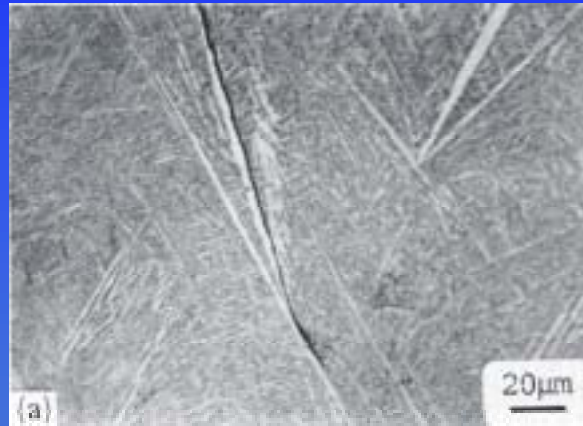


open symbols - coarse-grained
closed symbols - fine-grained



Fatigue Crack Nucleation Sites

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FH-WQ



RH-WQ

Ti 6242

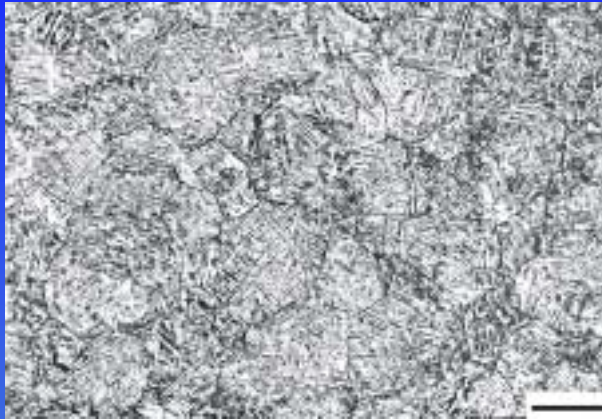


Fatigue Microcrack Propagation in Fine-Grained Ti-6Al-4V





Beta-Transformed (Diffusion-Controlled) Fine-Grained Microstructures



100μm



100μm

Alloy	Condition	A ₅ , %
Ti-6Al-4V	LG/AC	7.6
	FG/AC	11.4
Ti6242	LG/AC	6.1
	FG/AC	11.2
IMI 834	LG/AC	5.2
	FG/AC	14.4

Ti6242

Condition	$\sigma_{0.2}$, MPa	UTS, MPa	A ₅ , %	ϵ_F	$\epsilon_{pl.}$, %	σ_{max} 10 ⁷ , MPa	σ_{max} 10 ⁴ , MPa
FG/AC	930	1065	11	0.40	0.12	525	750
Bi-modal	945	1055	12	0.47	0.15	500	675



Graph showing Elongation (%) versus β -grain size (μm) for Ti-6Al-4V. The graph is divided into three regions based on the yield strength ($\sigma_{0.2}$):

- $\sigma_{0.2} < 1250 \text{ MPa}$ (Solid line)
- $\sigma_{0.2} = 1250-1300 \text{ MPa}$ (Dashed line)
- $\sigma_{0.2} > 1300 \text{ MPa}$ (Dotted line)

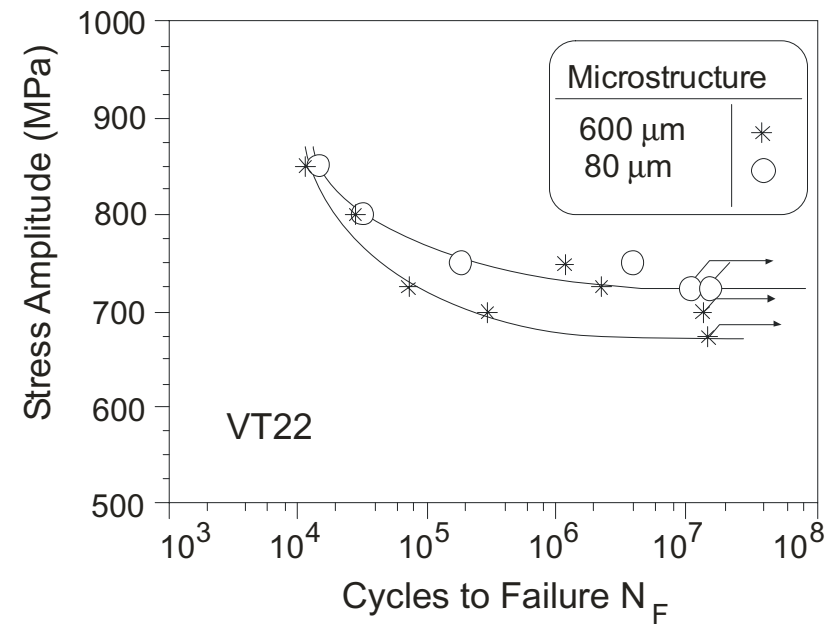
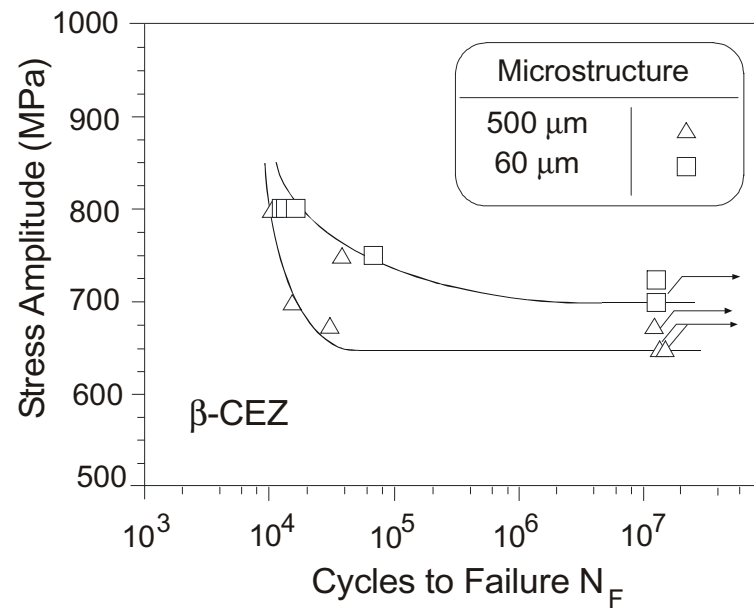
Data points are plotted for each region, showing a transition from high elongation to low elongation as grain size increases.



S-N Curves of β -CEZ and VT22

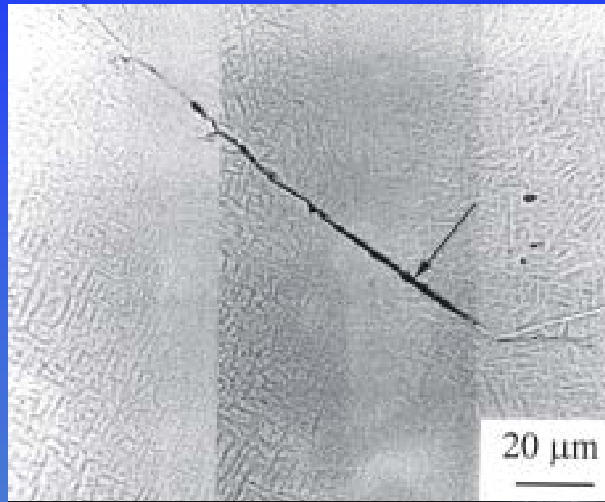
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room temperature, $R=-1$, 50 Hz

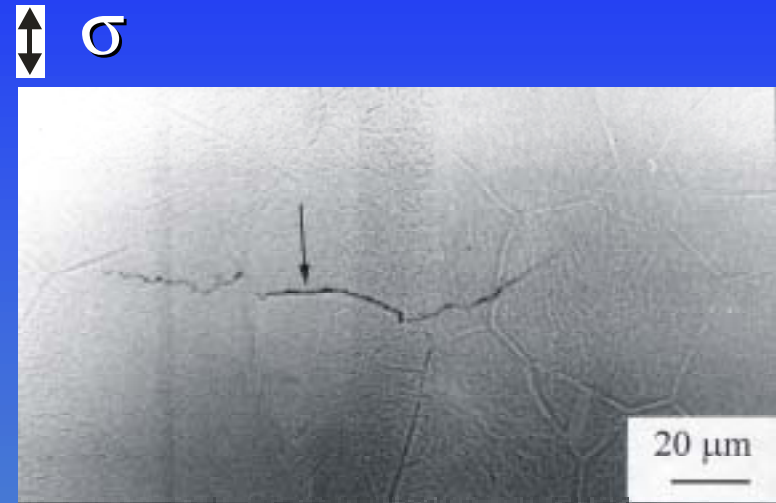




Fatigue Microcracks in β -CEZ



coarse-grained

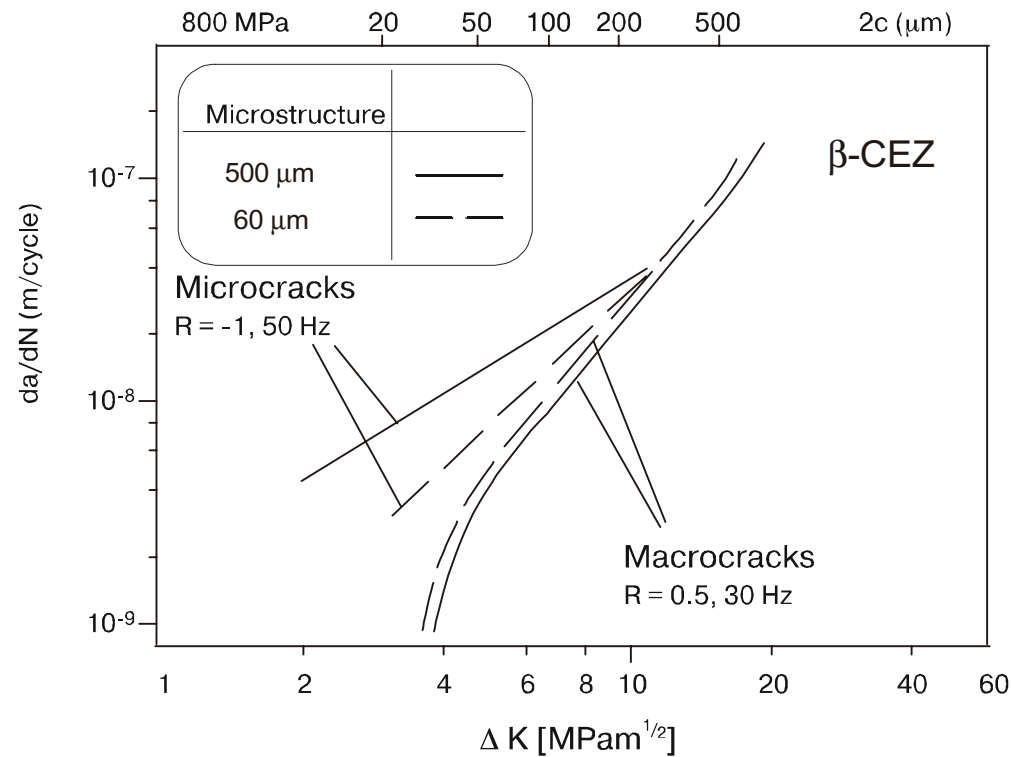


fine-grained



Micro- and Macrocrack Propagation in β -CEZ

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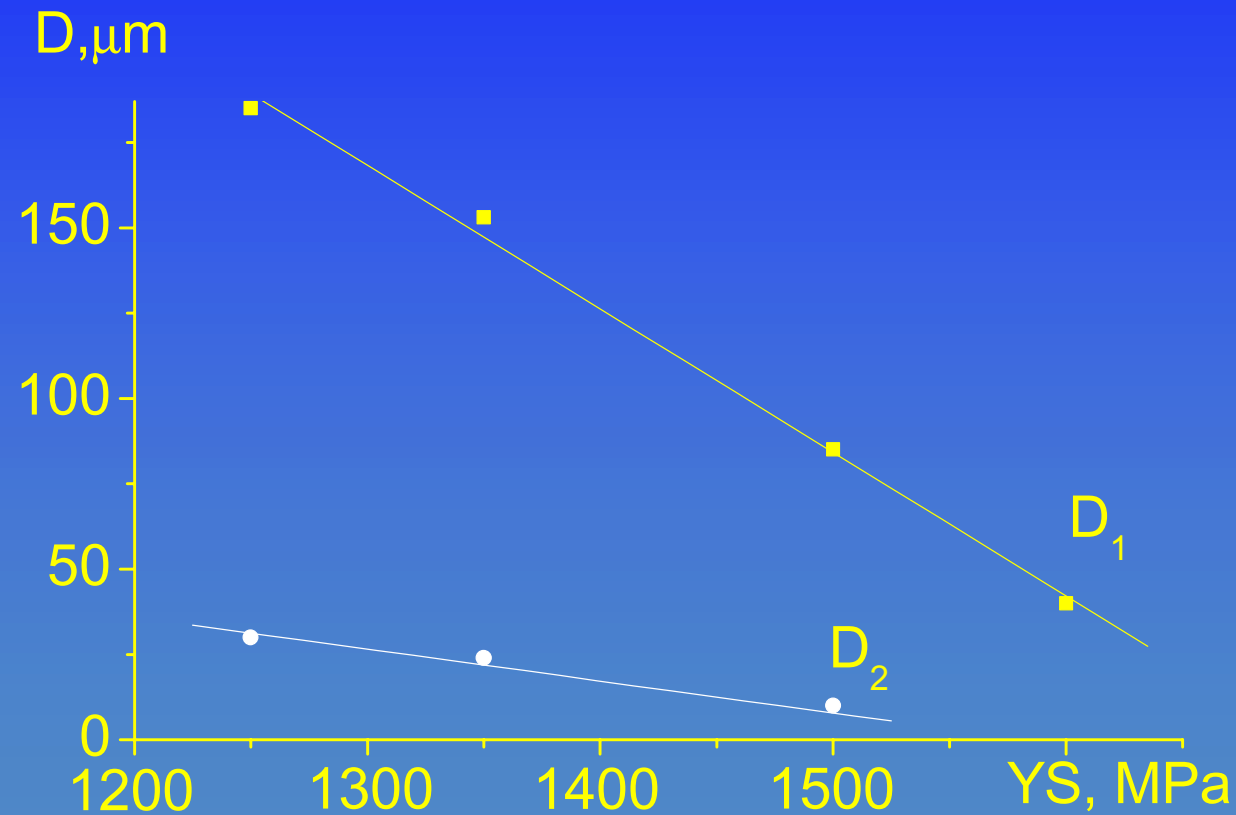


Super Strength Beta Alloys



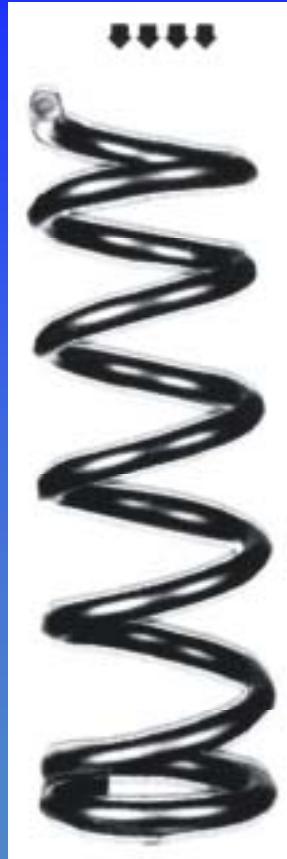
Critical Grain Sizes as a Function of Strength

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Coil Spring



$$\text{Volume} = \frac{G}{\tau^2} \left[2P^2 / R \right]$$

$$\text{Weight} = \frac{G\rho}{\tau^2} \left[2P^2 / R \right]$$

G – shear modulus

ρ – density

τ – shear stress

R – spring rate

P – load

Titanium advantage as compared to steels:

-25 % in volume

-2 to 1 in weight

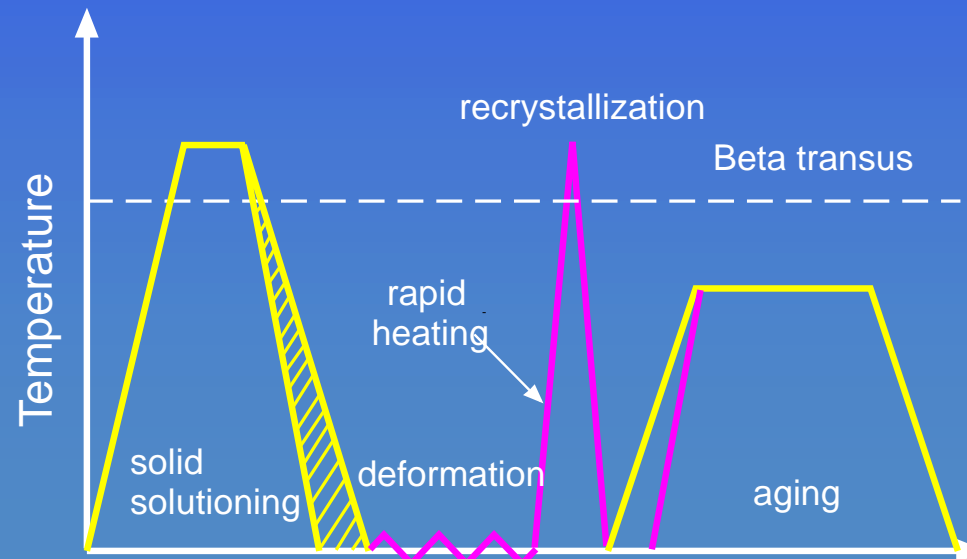


Cold Deformation in Thermal Strengthening

The introduction of cold deformation between solution treatment and aging may have a beneficial effect on the evolution of microstructure because dislocations and other defects can modify the nucleation of alpha precipitates.

Additional recrystallization heat treatment results in a finer grain microstructure that yields a better balance of strength and ductility.

The best way to control beta grain size during recrystallization is through the use of continuous, rather than isothermal, heat treatment.





Program Alloys

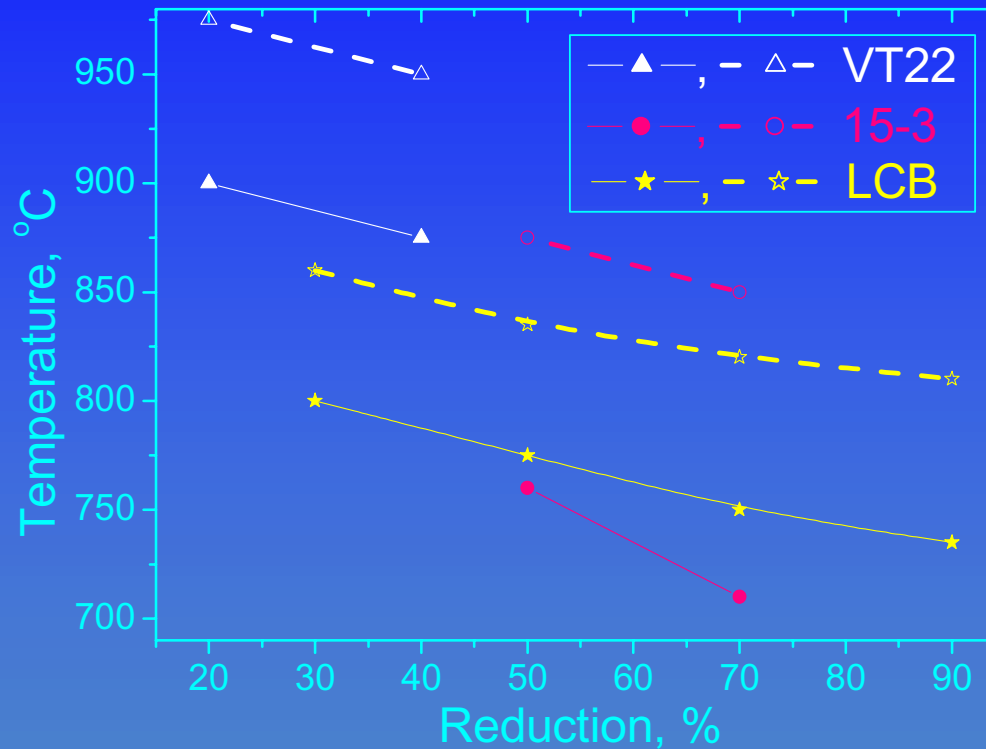
Alloy	Composition (wt. pct.)						Mo. Eq.	Beta Transus (°C)
	Al	Sn	Mo	V	Fe	Cr		
VT22	5.0	-	4.8	4.7	0.97	0.71	7.0	850
Ti-15-3	2.8	2.9	-	15.5	-	3.15	12.6	760
TIMETAL -LCB	1.5	-	6.8	-	4.5	-	18.4	790

Three commercial beta titanium alloys with different amounts of beta-stabilizing elements were used in the present program.



Recrystallization Behavior

Continuous Heating at 5 Ks^{-1}



Closed and open symbols correspond to the start and finish of recrystallization, respectively

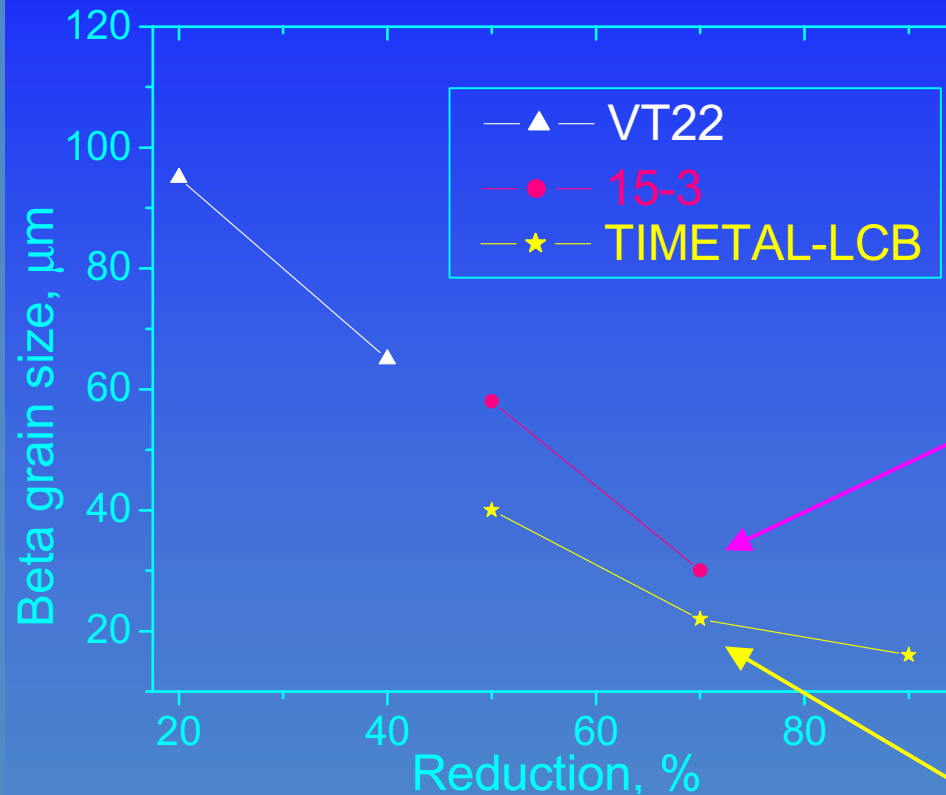
Ti METAL-LCB recrystallized more readily than Ti-15-3. The recrystallization interval was narrower and the recrystallization *finish* temperatures were lower for Ti METAL-LCB.



Recrystallization Behavior

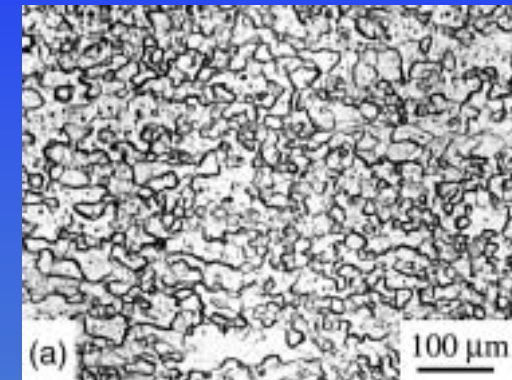
Continuous Heating at 5 Ks^{-1}

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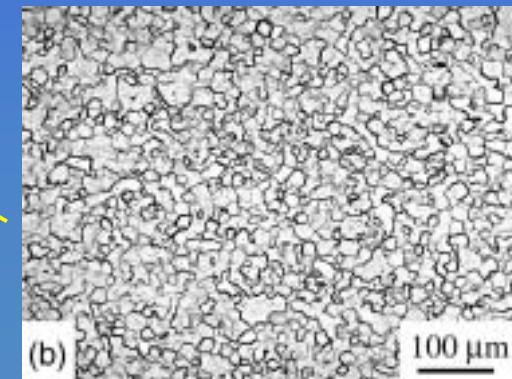


The narrower recrystallization interval and the lower recrystallization *finish* temperature led to a finer beta-grain size in TIMETAL-LCB for a given set of processing parameters.

Example:
70 pct. Reduction +
Continuous Heating



Ti-15-3



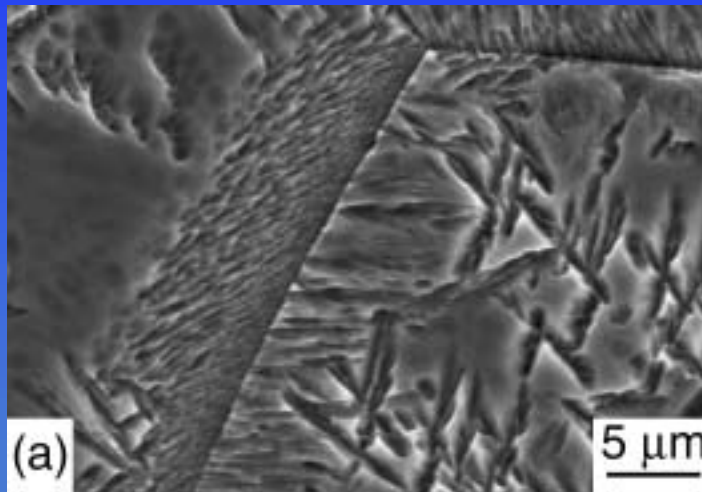
TIMETAL-LCB



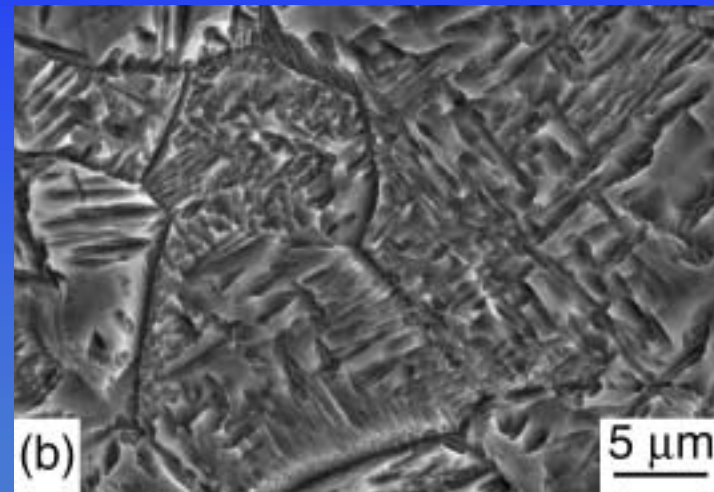
Aging Response

Microstructure

Ti-15-3 after aging at 560°C for 8 h



Coarse-grained



Fine-grained

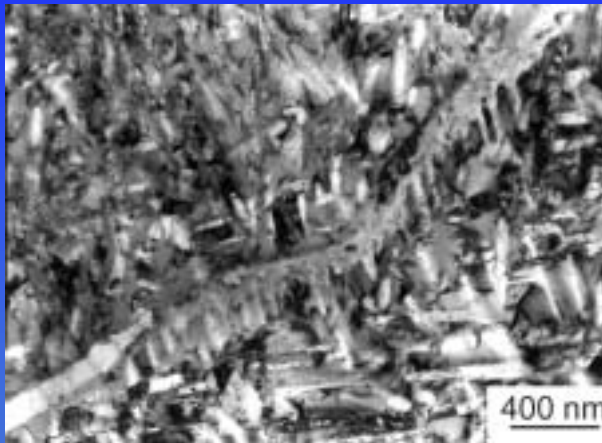
Alpha precipitation began earlier in samples with smaller beta grains and resulted in larger amounts of precipitate and noticeably fewer precipitate-free zones.



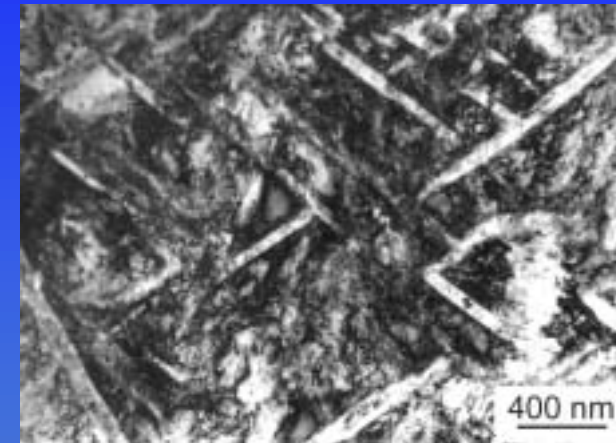
Intragrain Microstructure in TIMETAL[®]-LCB

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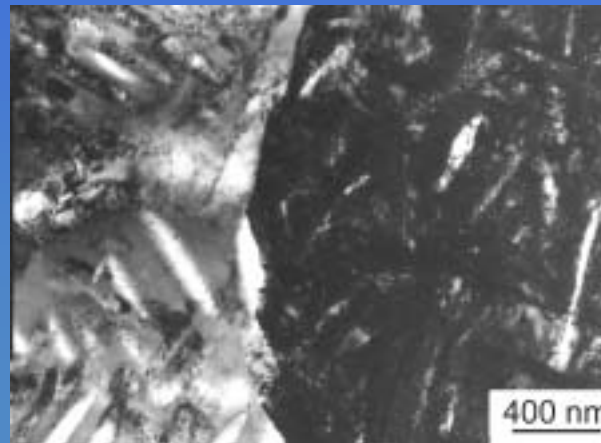
Influence of aging time, $T=560^{\circ}\text{C}$



2 h



4 h



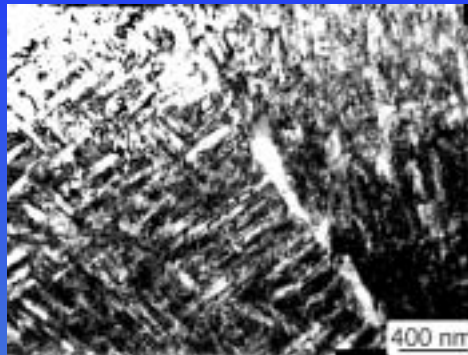
8 h



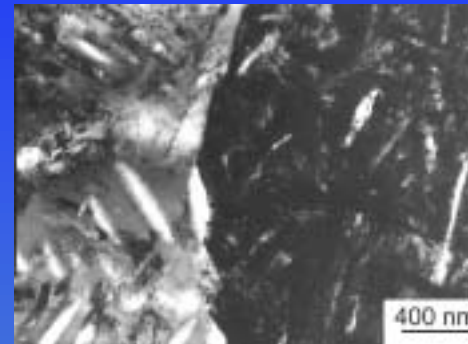
Intragrain Microstructure in TIMETAL[®]-LCB

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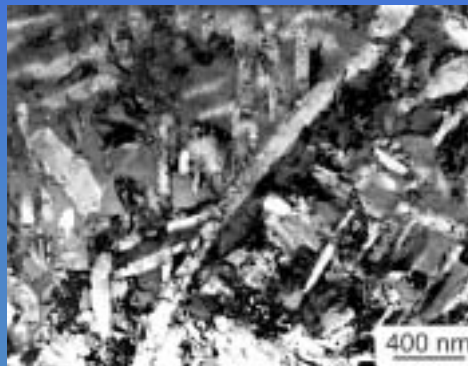
Influence of aging temperature, $t=8$ h



538 °C



560 °C



580 °C



600 °C



Mechanical Properties of LCB: Influence of Grain Size

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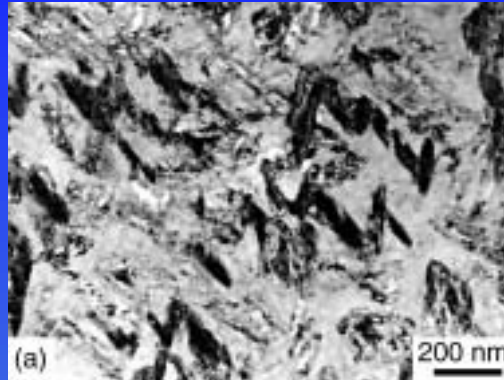
#	Solid solutioning	Grain size, μm	Reduction, %	Recrystallization	Grain size, μm	YS, MPa	UTS, MPa	A ₅ , %	RA, %
1	β -furnace	90	—	—	90	1210	1210	brittle failure	
2	β -furnace	90	50	RRA	30	1395	1425	6.8	24
3	β -rapid heating	20	—	—	17	1450	1460	5.0	19
4	β -rapid heating	20	50	RRA	12	1420	1470	8.2	22.5
5	β -rapid heating	20	70	RRA	7	1475	1490	10.3	25.5

Aging: 538 °C, 8h



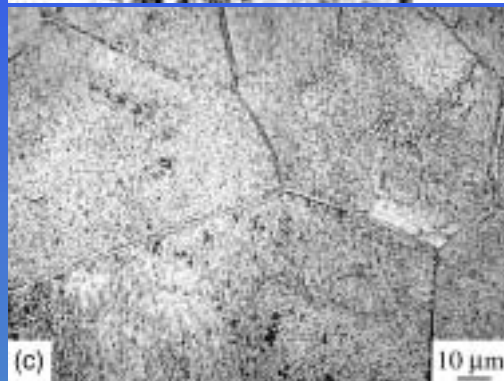
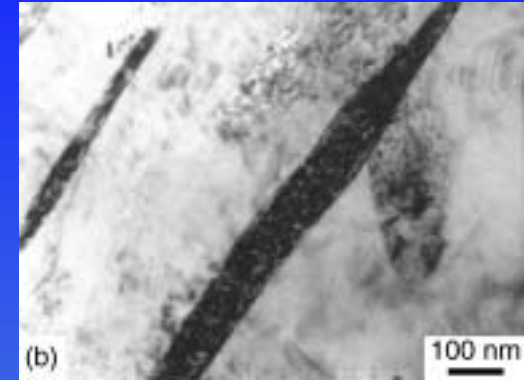
Aging Response

Influence of Heating Rate



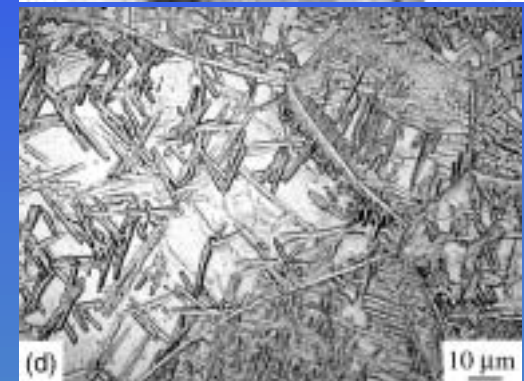
TIMETAL-LCB

538°C, 2 h



676 °C, 75 min

0.033 Ks⁻¹ 20 Ks⁻¹

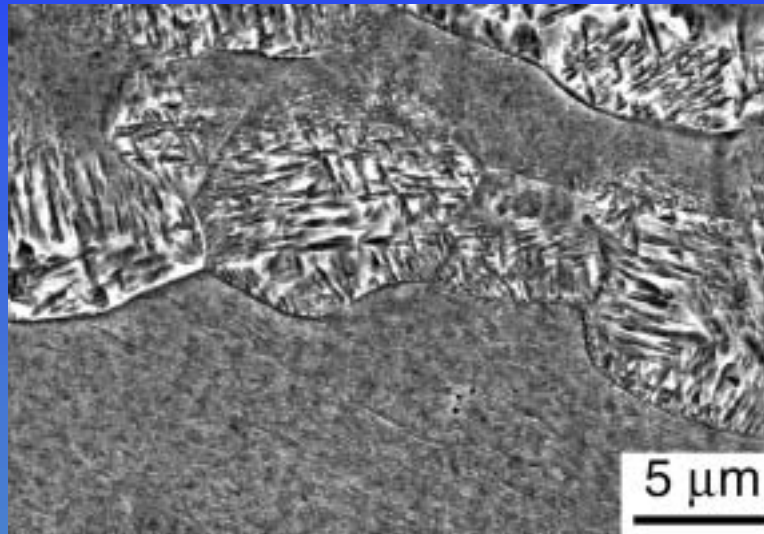


With slow heating, omega precipitation preceded nucleation of the alpha phase and resulted in a very fine and uniform microstructure. Rapid heating depressed the intermediate stages of decomposition resulting in the formation of a nonuniform, coarse structure of alpha, apparently generated via a shear mechanism. The only difference was that the transition between the two precipitation mechanisms occurred at a much slower heating rate due to the generally slower precipitation kinetics.



Aging Response Microstructure

The aged microstructure was greatly affected by residual deformation substructure if recrystallization was nonuniform such as in the case of low cold reductions.



partially recrystallized Ti-15-3
after aging at 538°C for 4 h

Alpha precipitation was markedly different in the recrystallized and the recovered areas. The much finer precipitate structure in recovered regions suggests that continuous heating into the recovery-temperature range may provide a substantially-improved balance of mechanical properties in beta-titanium alloys.



Modulated Structure of Alpha Double-Prime Martensite

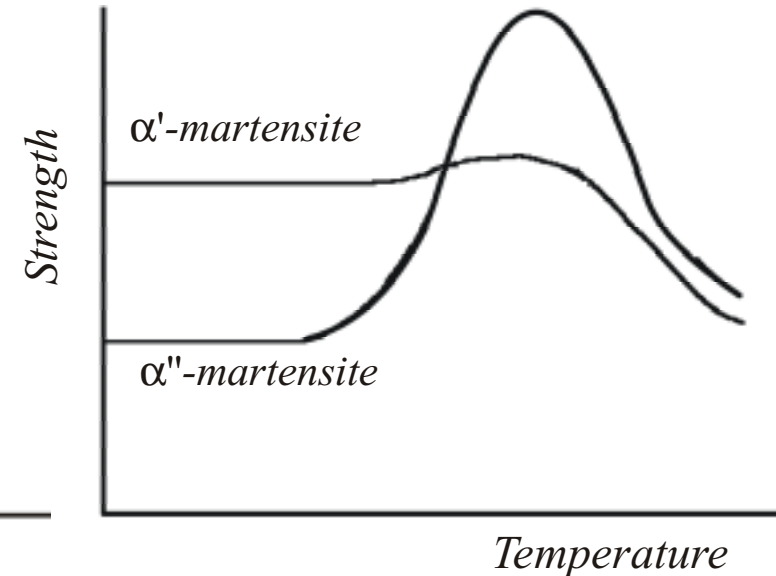
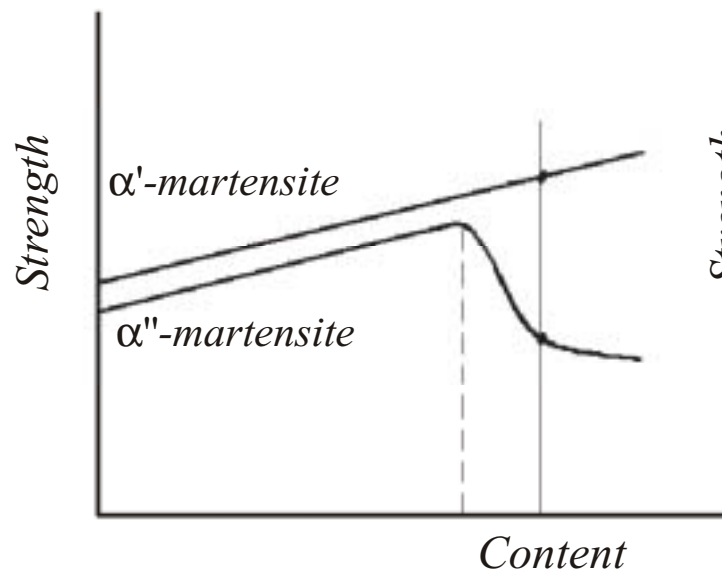


Strength of Titanium Martensite with Different Crystal Structure

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Concentrational dependence

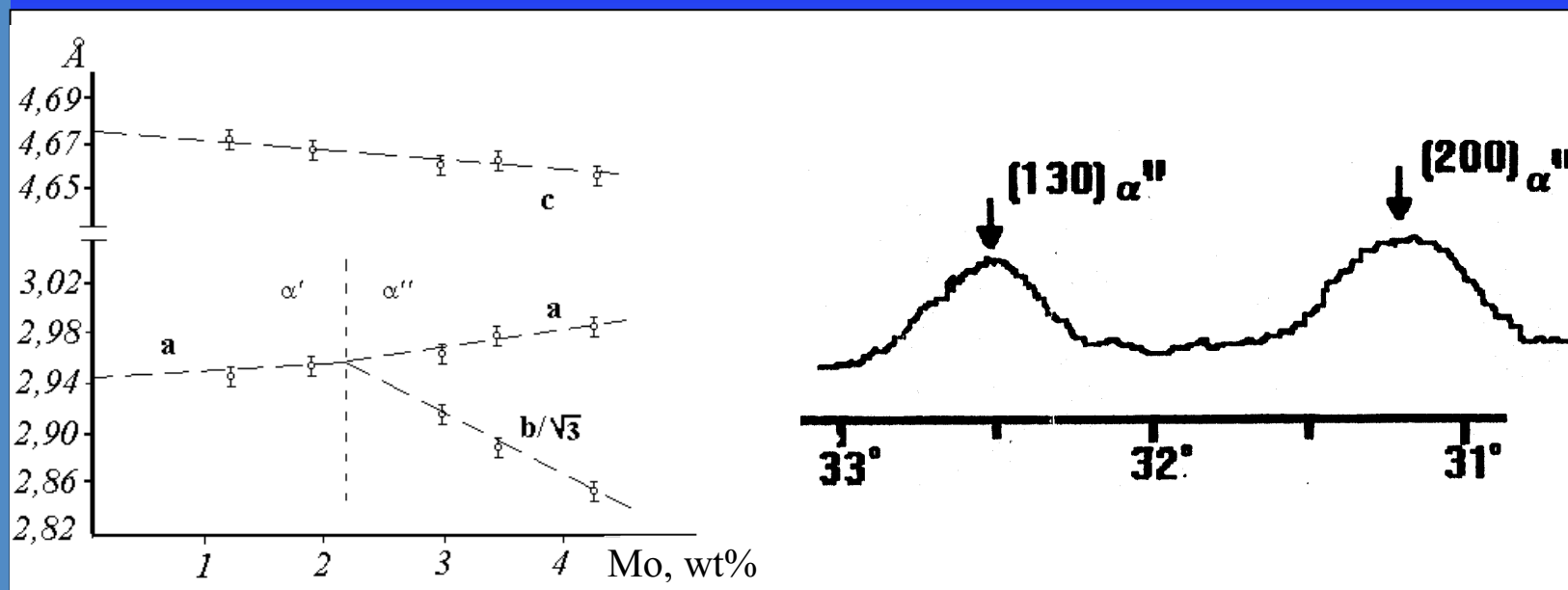
Temperature dependence





Lattice Parameters of α'' -Martensite

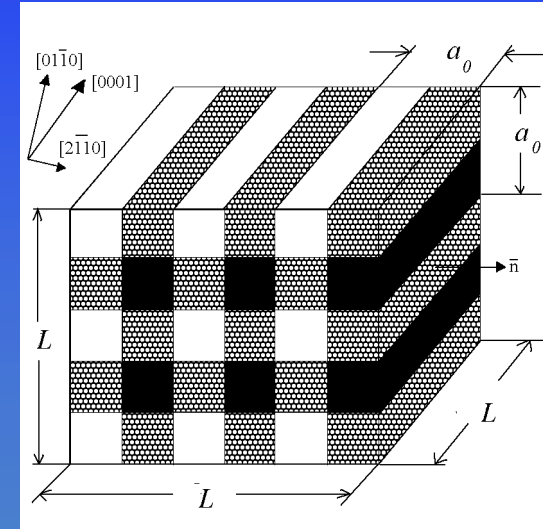
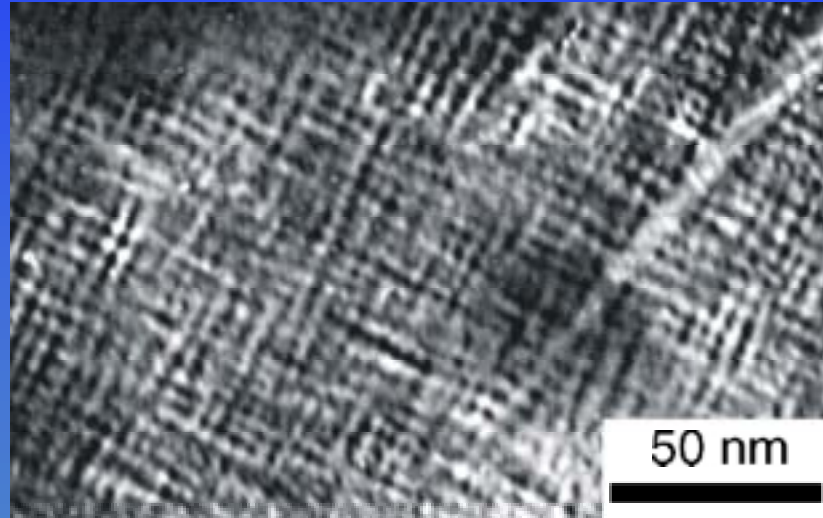
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Two Dimensional Modulated Structure in h.c.p. Solid Solution

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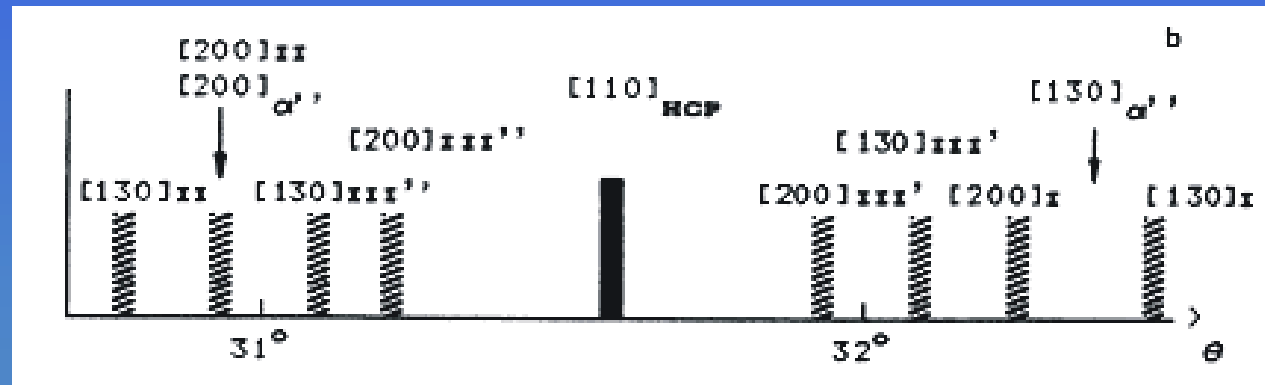


Diffraction Effects of Modulated Structure

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Deformation under the action of coherent stresses

$$\varepsilon_{ij}(\mathbf{r}) = Ku_0 \left[\begin{pmatrix} n_1^2 & n_1 n_2 & 0 \\ n_1 n_2 & n_2^2 & 0 \\ 0 & 0 & 0 \end{pmatrix} C^{[n_1 n_2 0]} + \begin{pmatrix} n_1^2 & -n_1 n_2 & 0 \\ -n_1 n_2 & n_2^2 & 0 \\ 0 & 0 & 0 \end{pmatrix} C^{[n_1 \bar{n}_2 0]} \right]$$





Thermodynamic Analysis of h.c.p. Solid Solutions

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Full mixing energy

$$W_{pp'}(\mathbf{r}) \equiv W_{pp'}^{MeMe}(\mathbf{r}) + W_{pp'}^{TiTi}(\mathbf{r}) - 2W_{pp'}^{MeTi}(\mathbf{r})$$

Deformation part

$$\tilde{V}_{pp'}^{MeMe}(\mathbf{k}) \approx -\tilde{F}_{\lambda p}^{i*}(\mathbf{k}) \tilde{G}_{ij}^{\lambda\mu}(\mathbf{k}) \tilde{F}_{\mu p'}^j(\mathbf{k}) + Q_p \delta', \quad (\mathbf{k} \neq \mathbf{0})$$

$$\tilde{V}_{pp'}^{MeMe}(\mathbf{0}) \approx -v_{u.c} \sigma^{pim} L_{p'im} + Q_p \delta_{pp'}$$

Electrochemical part

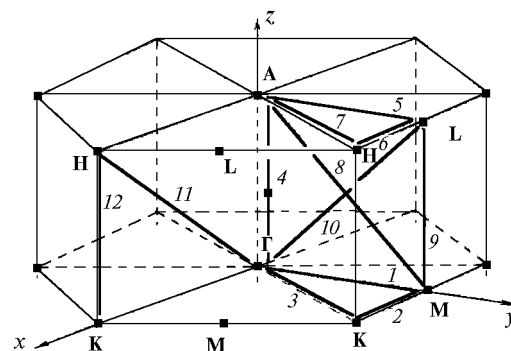
$$\tilde{\varphi}_{pp'}^{MeMe}(\mathbf{k}) = \sum_{\mathbf{R}} \varphi_{pp'}^{MeMe}(\mathbf{R}) \exp(-i\mathbf{k} \cdot \mathbf{R}).$$

$$\varphi_{pp'}^{MeMe}(\mathbf{r}) \approx (A_1 A_2 \exp(-(b_1 + b_2)\mathbf{r}))^{1/2}$$

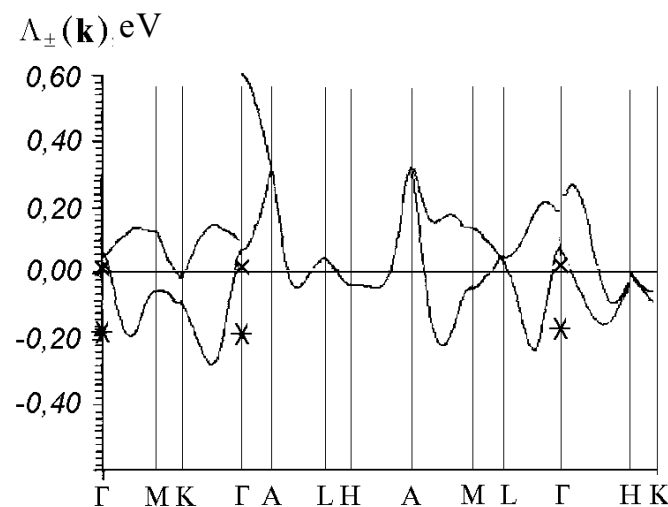


Fourier Components of Full Mixing Energy

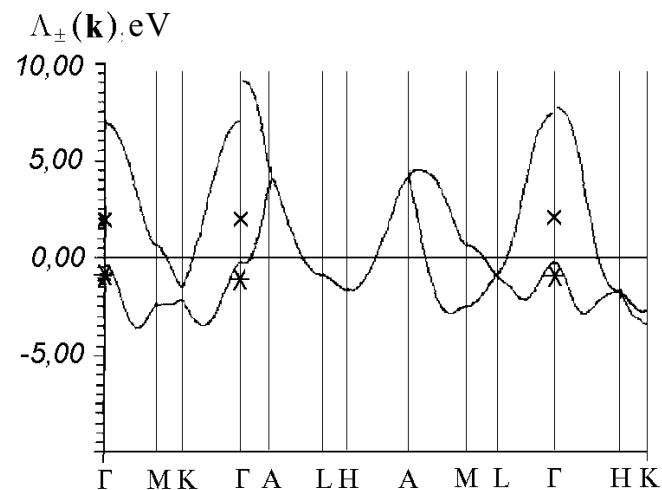
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Ti-Fe



Ti-Mo

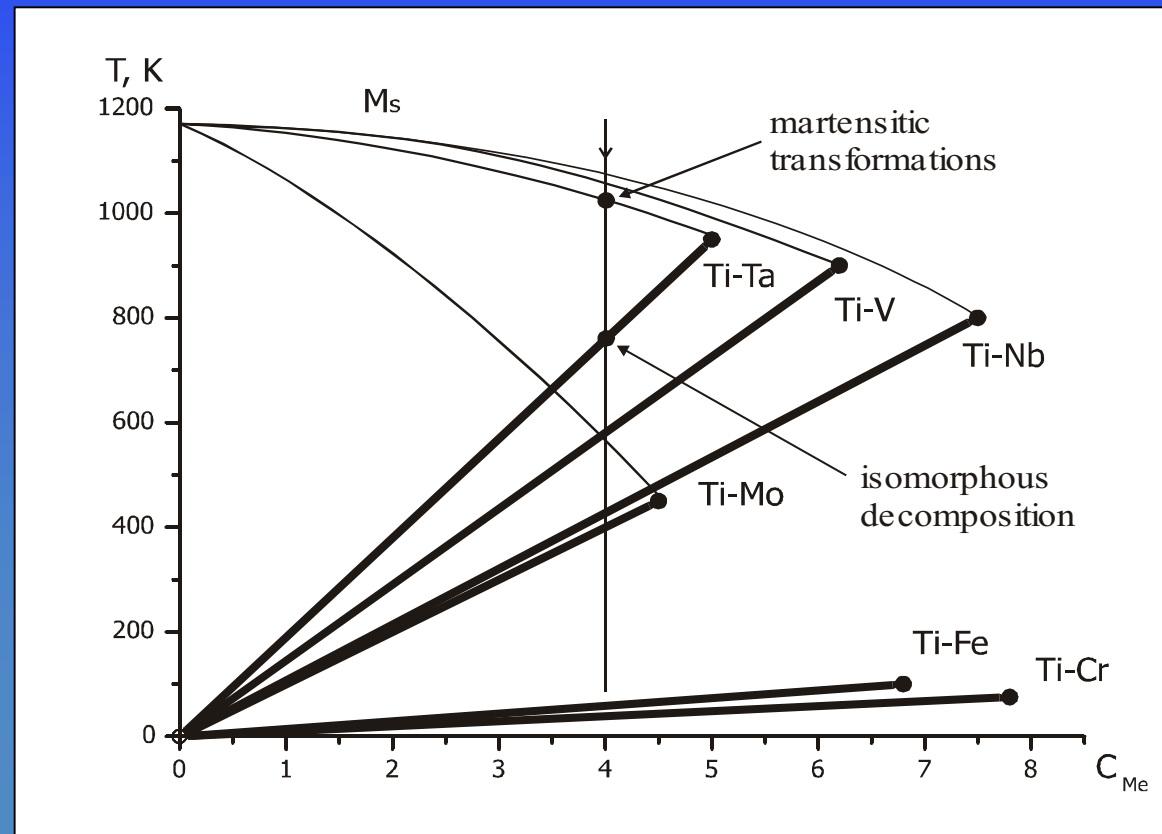




Stability of h.c.p. Solid Solutions

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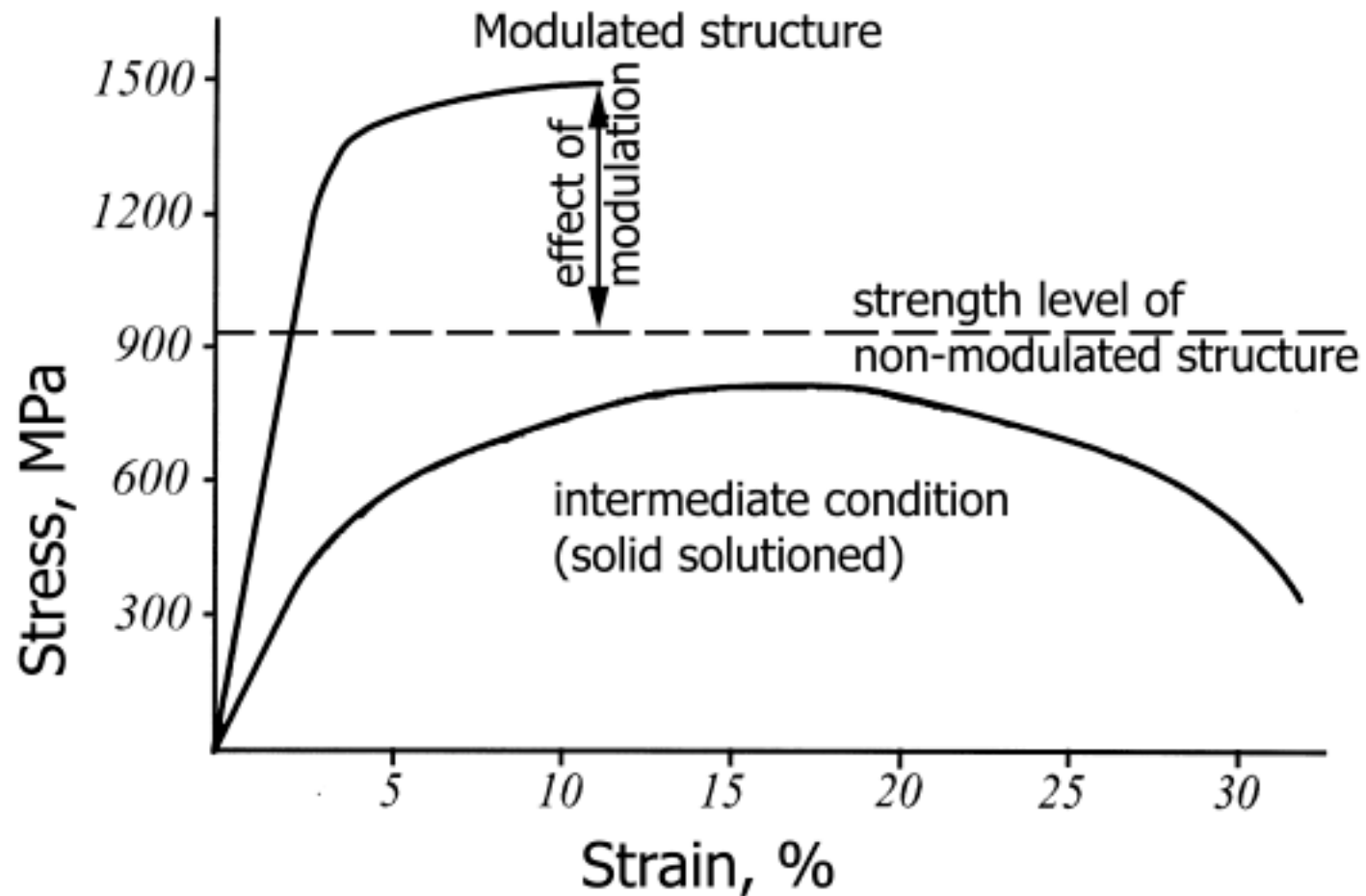
$$b(\mathbf{k}, T, c) = \begin{vmatrix} \tilde{V}_{11}(\mathbf{k}) + \frac{k_B T}{c(1-c)} & \tilde{V}_{12}(\mathbf{k}) \\ \tilde{V}_{21}^*(\mathbf{k}) & \tilde{V}_{22}(\mathbf{k}) + \frac{k_B T}{c(1-c)} \end{vmatrix} \geq 0$$





Tensile Curves of Modulated Structures

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Texture Controlled Grain Growth Kinetics at Continuous Heating



Grain Growth upon Continuous Heating

Isothermal conditions:

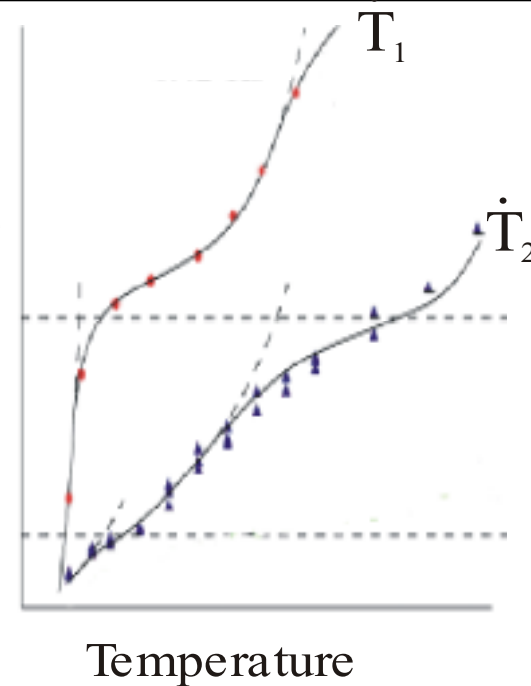
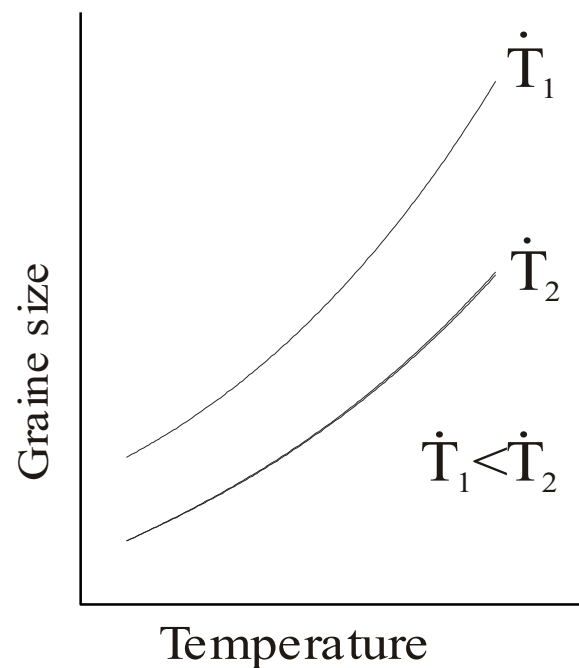
$$D^n - D_0^n = Kt \exp(-Q/RT)$$

Continuous heating conditions

$$D^n - D_0^n = (KR/\dot{T}Q) \left\{ \left[T_f^2 \exp(-Q/RT_f) \right] - \left[T_i^2 \exp(-Q/RT_i) \right] \right\}$$

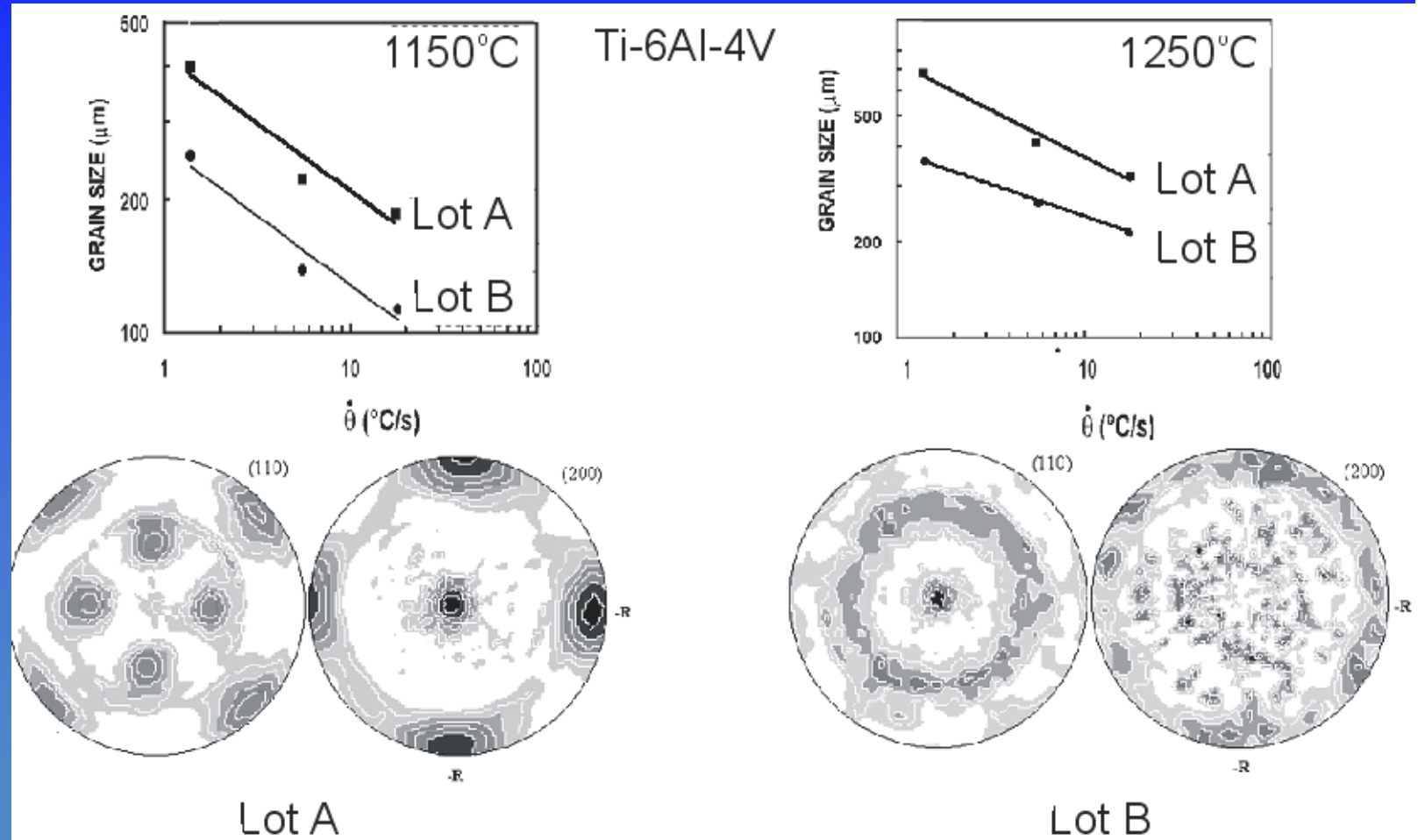
Isotropic condition

Textured condition





Influence of Texture on Beta Grain Size¹



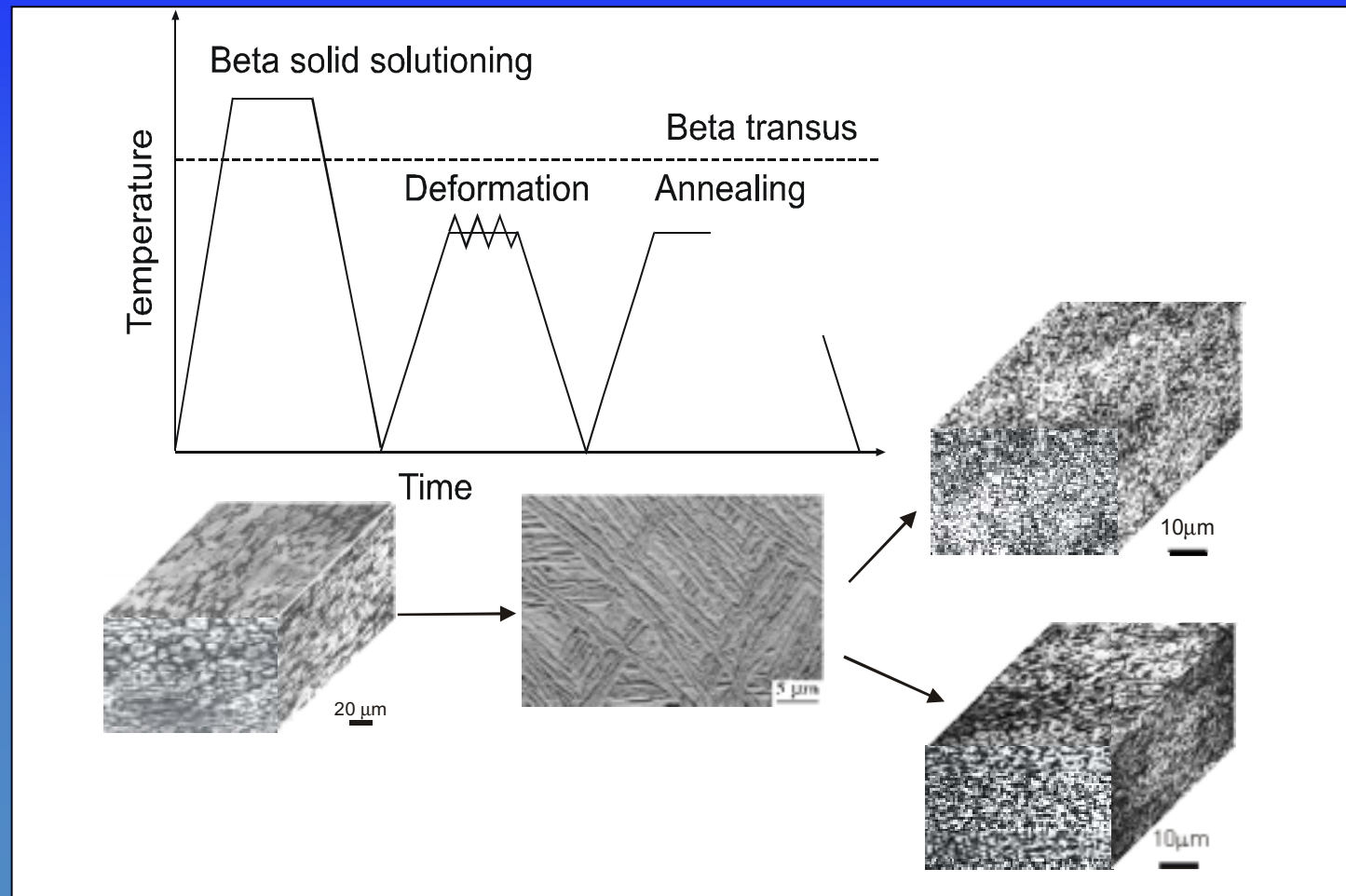
¹ S. L. Semiatin, P. N. Fagin, M. G. Glavicic, I. M. Sukonnik, O. M. Ivasishin
Mater. Sci. Eng. A299, 2001, 225



Preparation of Starting Materials

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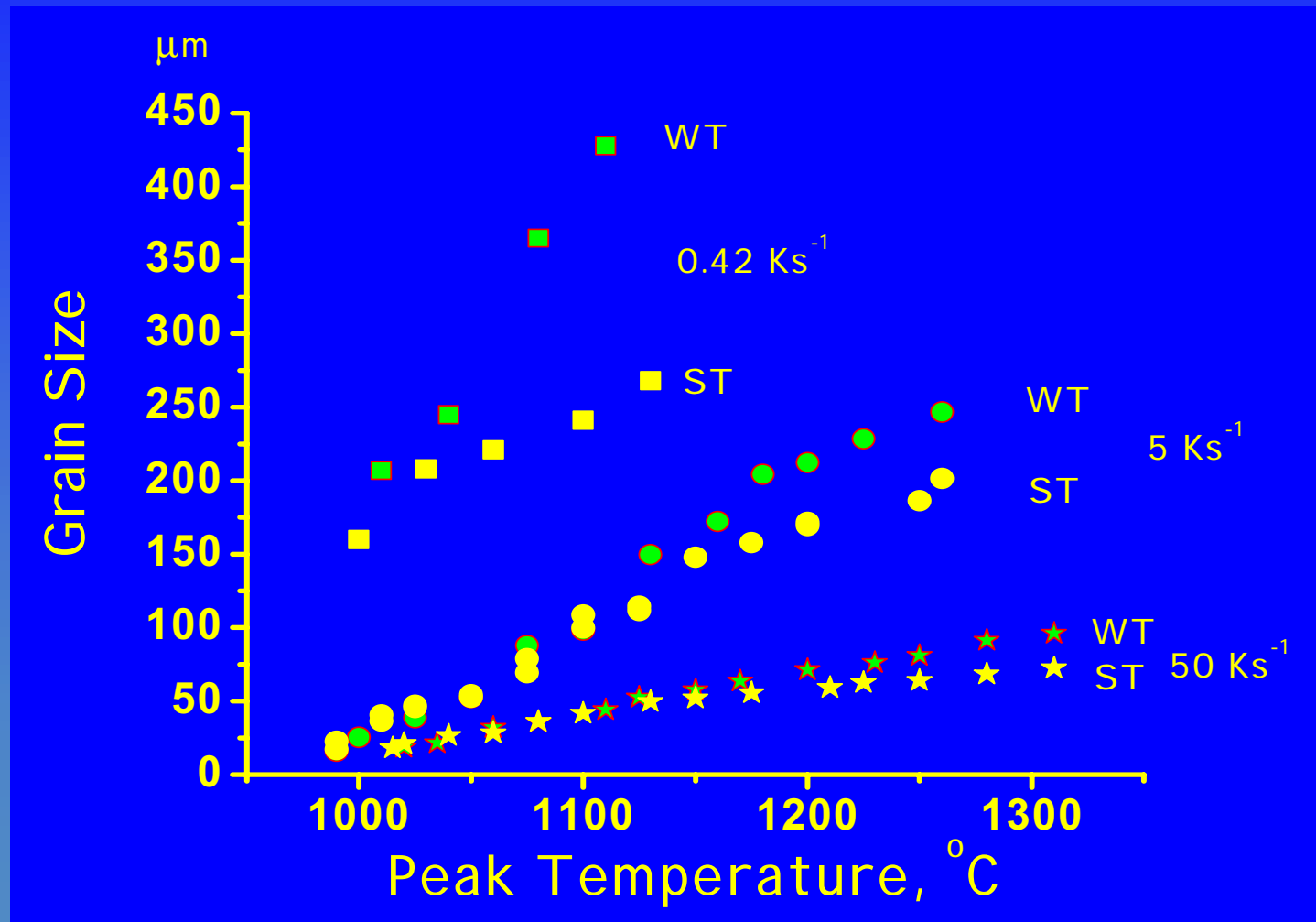
Ti-6Al-4V: Ti-6,05Al-4.40V-0.15Fe





Beta Grain Growth Kinetics for the Ti-6Al-4V Alloy

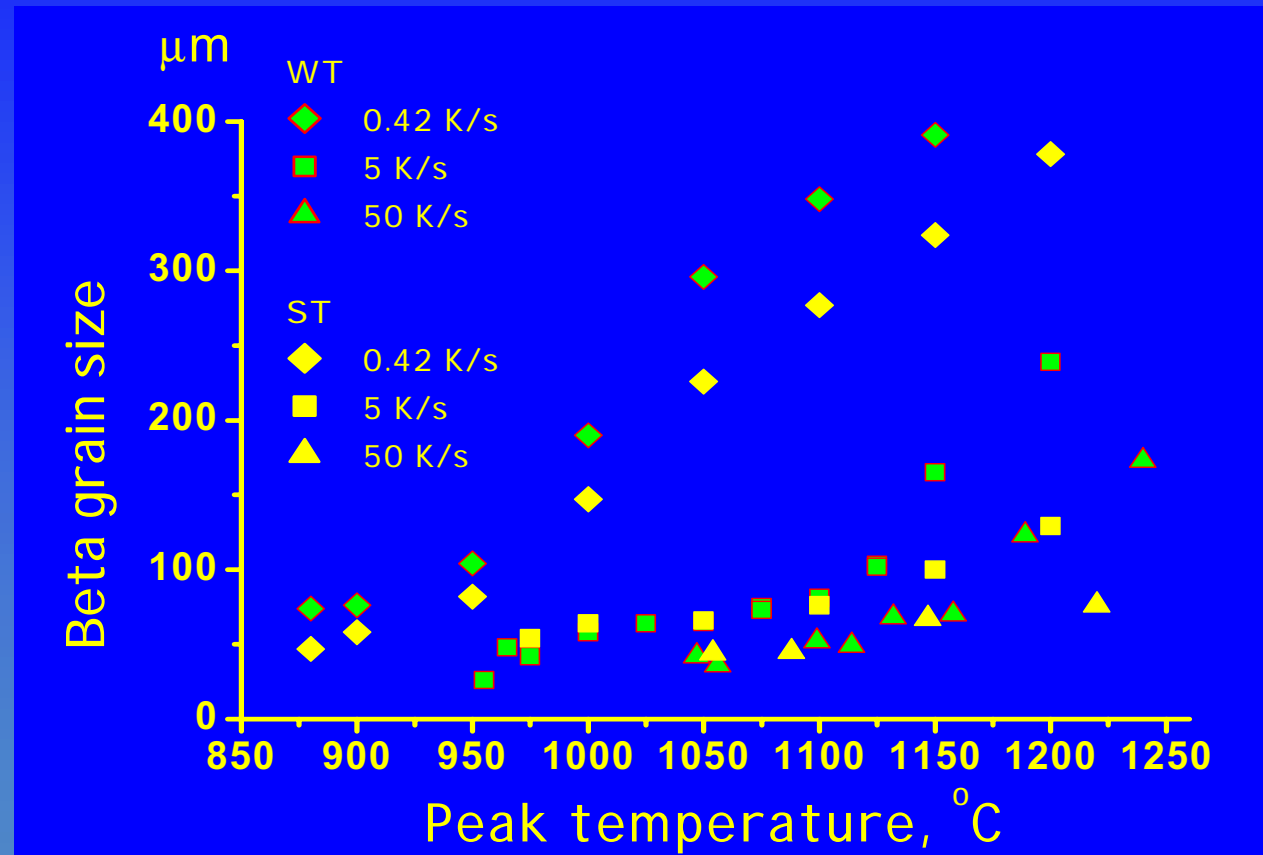
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Beta Grain Growth Kinetics for the VT16 Alloy

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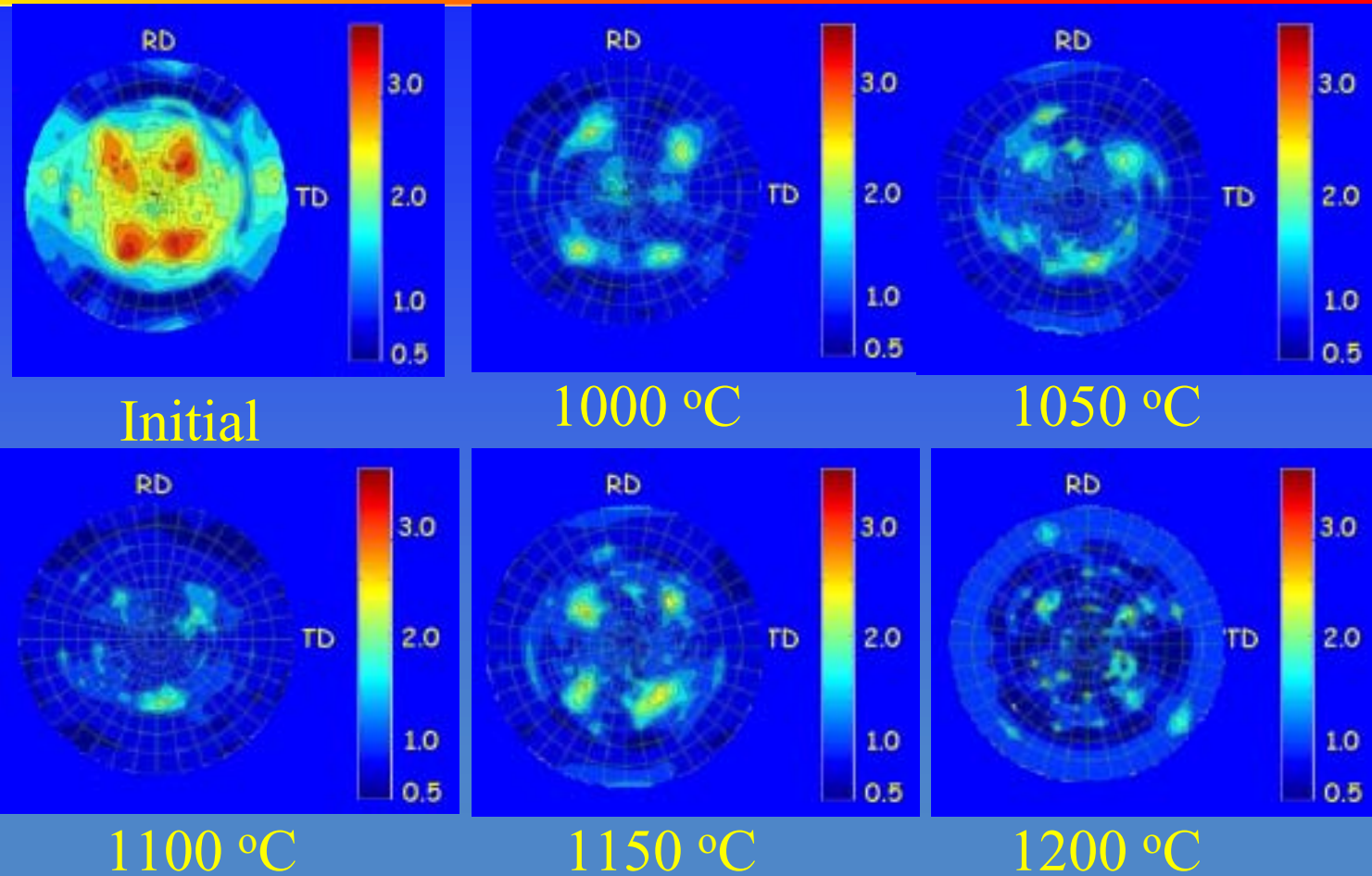
WT - weak textured condition

ST - strong textured condition



Cyclic Texture Evolution in Ti-6Al-4V Alloy Leading to Discontinuous Grain Growth

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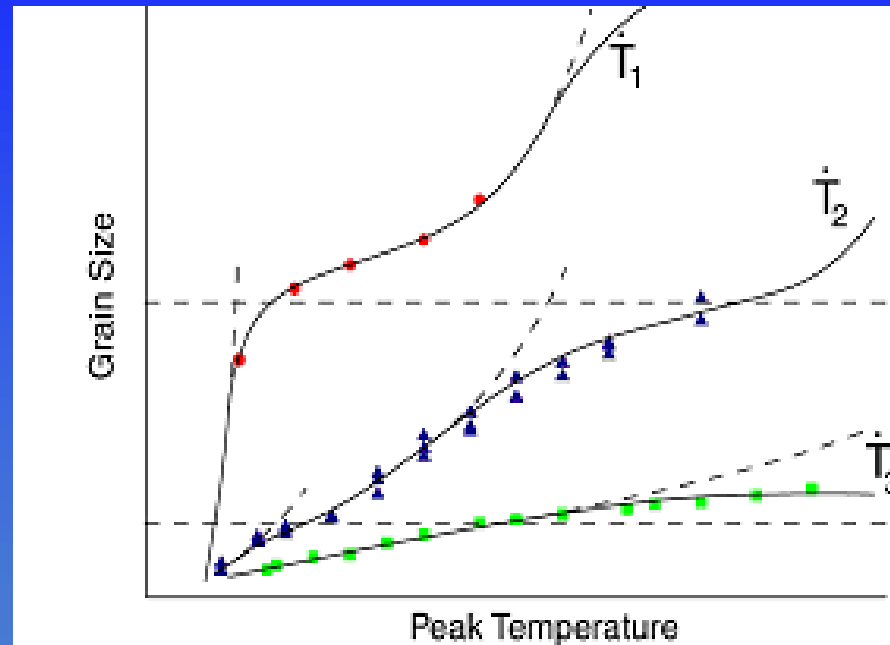


During continuous heating, the texture of titanium alloys undergo a cyclic evolution leading to alternating stages of fast and slow grain growth



Typical Beta Grain Growth Behavior Based on Experimental Data

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The discontinuous character of grain growth observed for the strong textured material, for which periods of slow and fast grain growth alternates are the result of the periodic texture evolution.



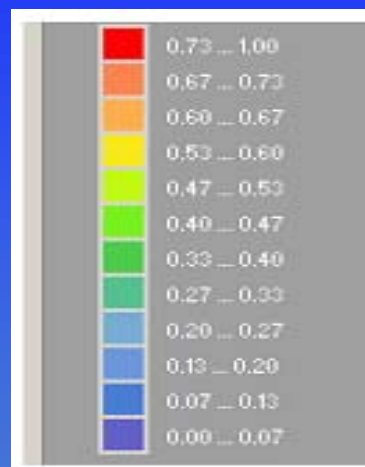
Mobility of Boundaries Depending on Texture

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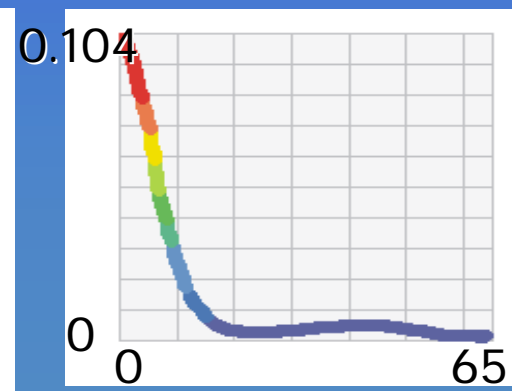
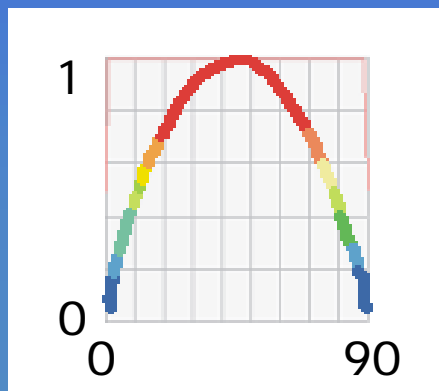
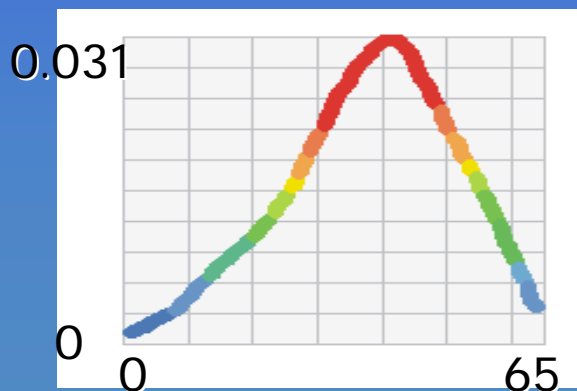
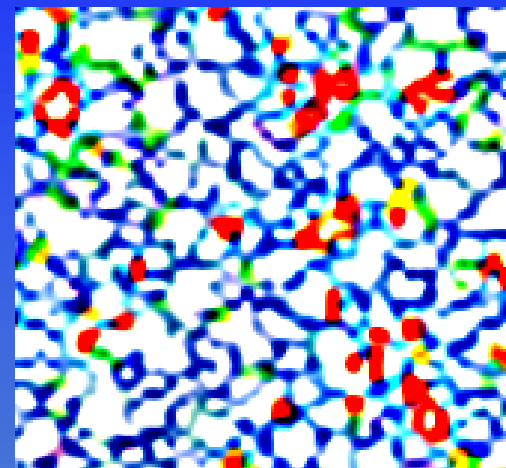
Not textured material



Relative GB mobility
scale



Textured material

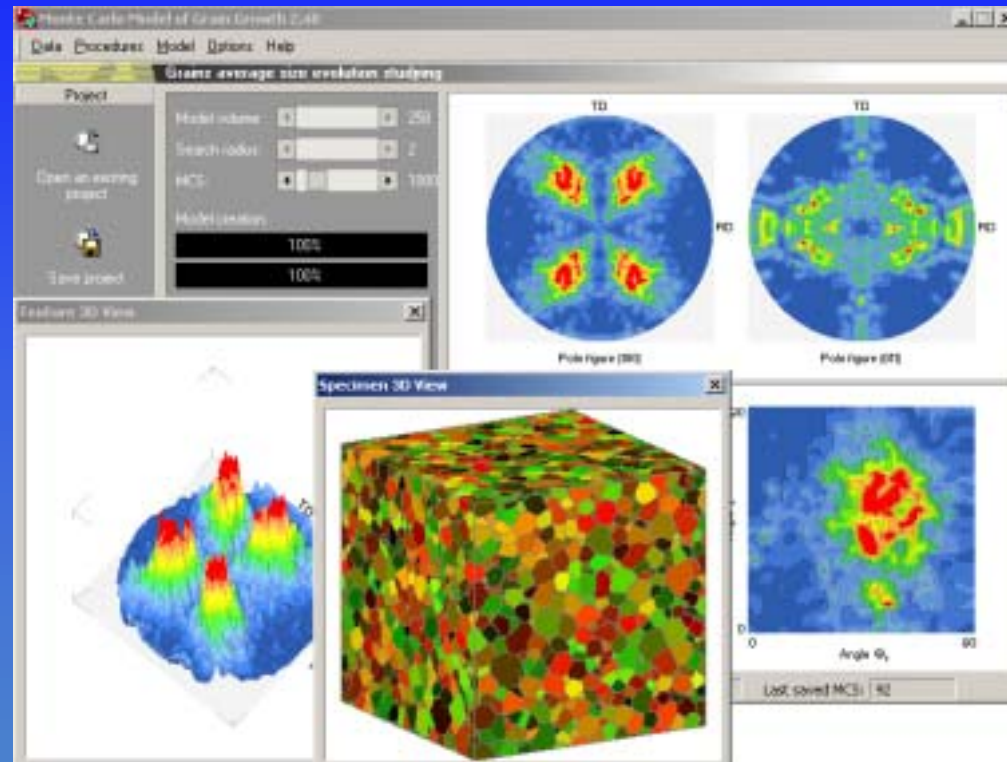


GB distribution functions and GB relative mobility vs. GB misorientation



3D Monte Carlo Simulation of Texture Controlled Grain Growth

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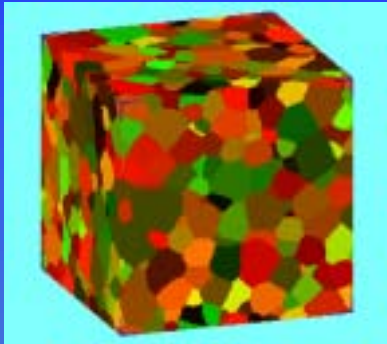


Program complex based on for computationally-efficient 3D Monte-Carlo algorithm was developed to quantify the interaction of grain growth and texture development during the beta annealing of titanium alloys

Outputs quantified the evolving texture in terms of pole figures or crystallite orientation distribution functions and statistics on the grain structure such as the grain-size distribution and grain boundary misorientation distribution function.



MC Simulation Features



Metropolis orientation flip probability:

$$W = \begin{cases} M_{ij} \exp(-\Delta G / k_b \mathbf{T}); & \Delta G > 0, \\ M_{ij} & \Delta G \leq 0, \end{cases}$$

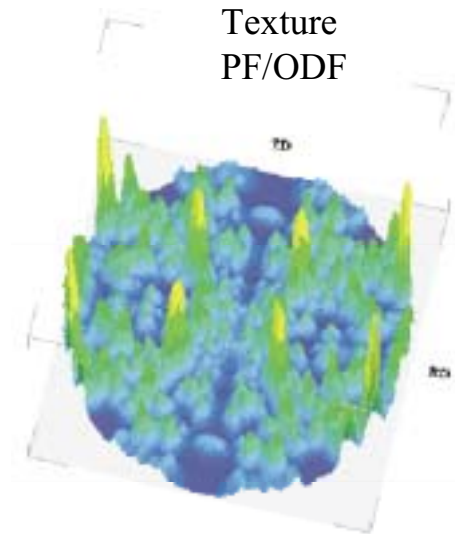
Misorientation dependent GB mobility:

$$M_{ij} = M_0 \quad V(\mathbf{g}_i, \mathbf{g}_j) = M_0 \quad V(\epsilon).$$

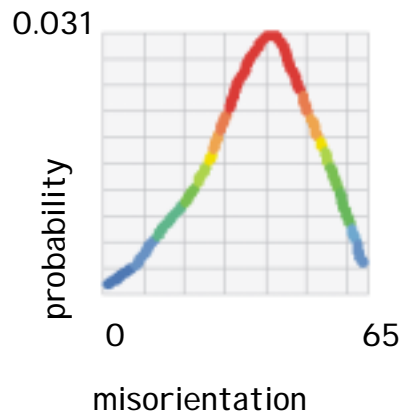
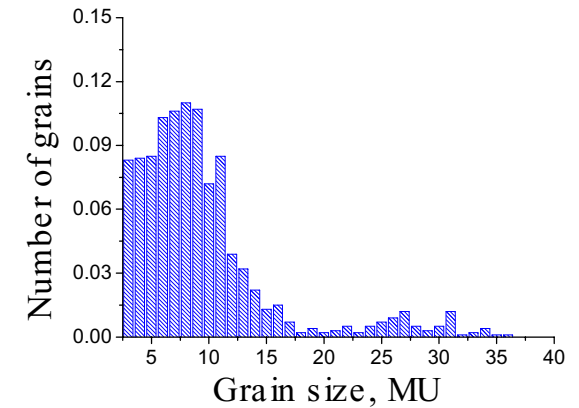
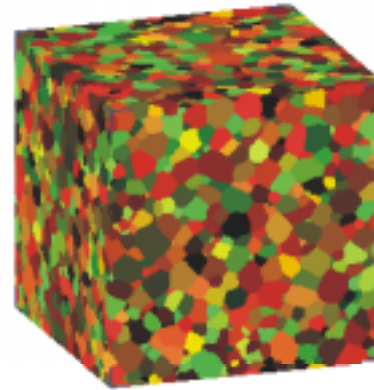


MC Simulation Features

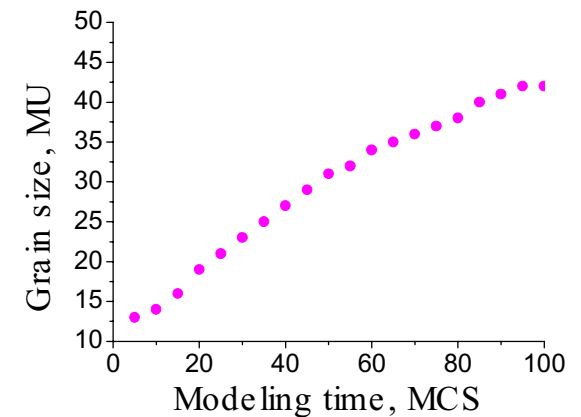
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modeling volume 250^3 MUs
up to 200 000 grains
up to 728 000 orientations



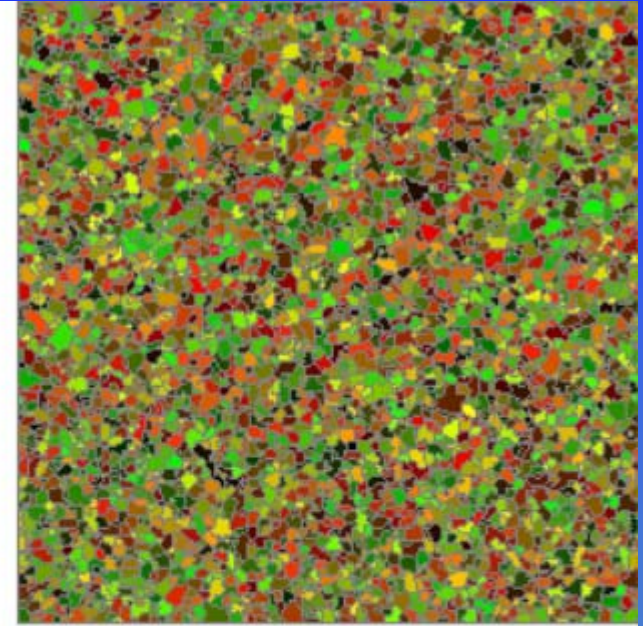
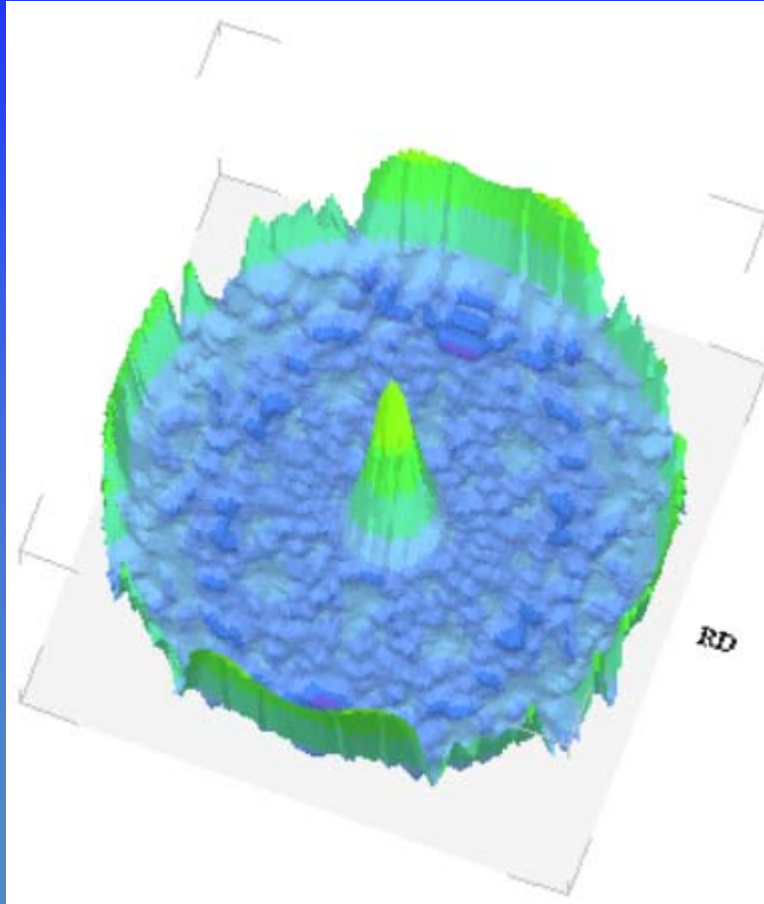
Outputs quantified the evolving texture in terms of pole figures or crystallite orientation distribution functions and statistics on the grain structure such as the grain-size distribution and grain boundary misorientation distribution function.





Texture Evolution During Grain Growth – the 3D MC Modeling

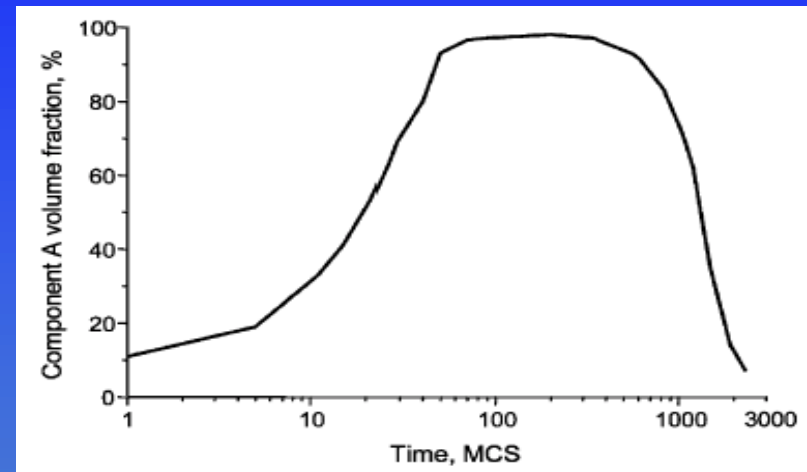
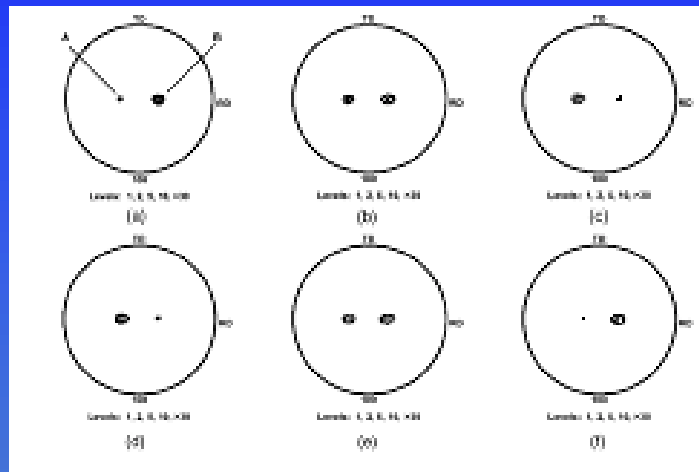
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MC Modeling: Grain Growth Kinetics in Two-Component Textured Material

Cyclic texture evolution and volume fraction of the texture component A

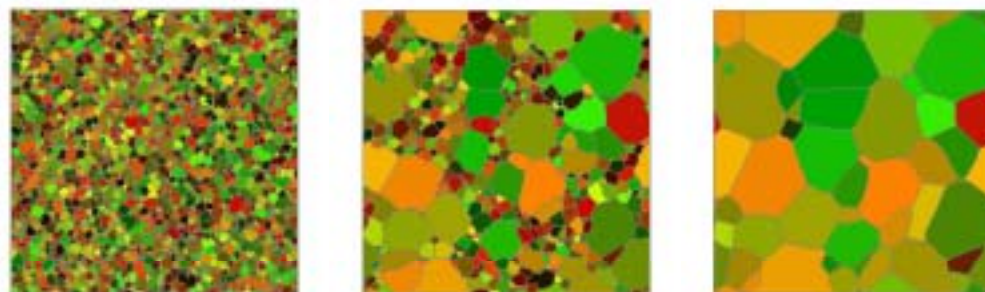


Kinetics of the average grain size in the modeling volume: periods of slow and fast grain growth are the result of the periodic texture evolution

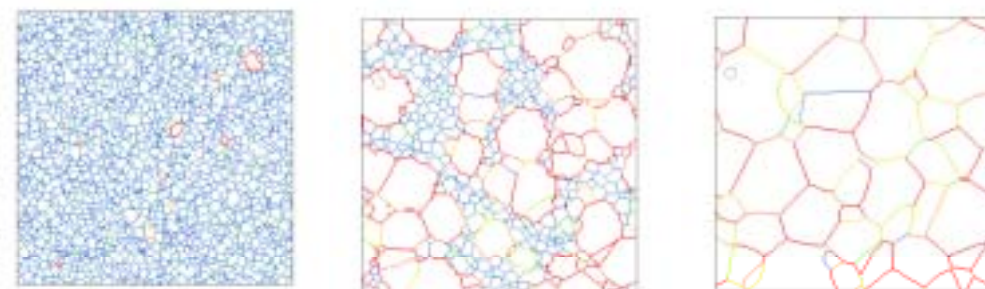


MC Modeling: Abnormal Grain Growth in Heavily Textured Material

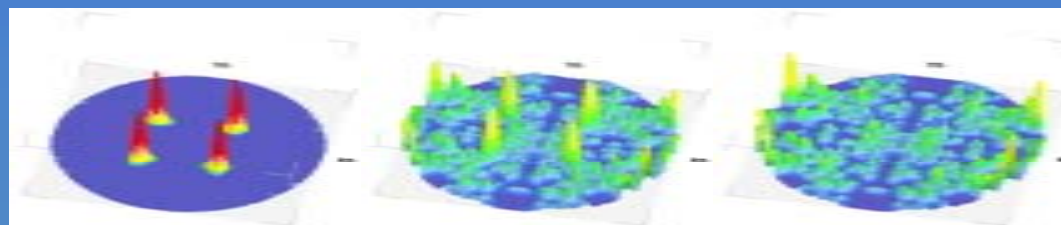
Microstructure



GB mobility



Texture



Time, MCS

010

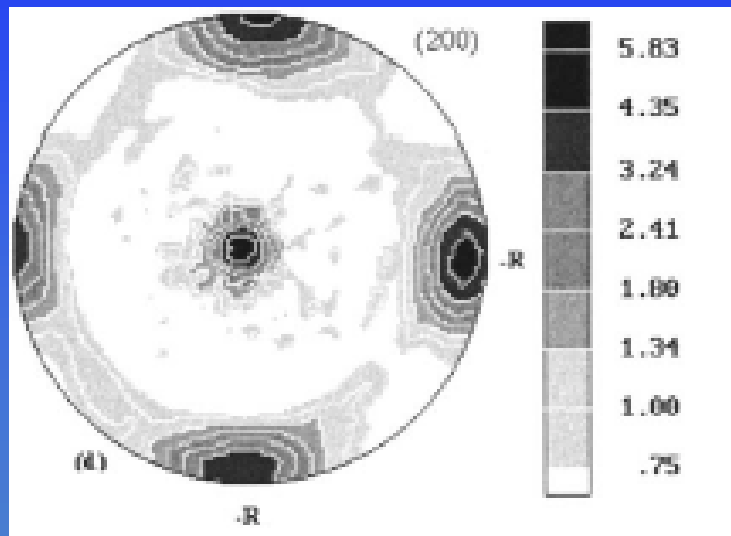
100

250

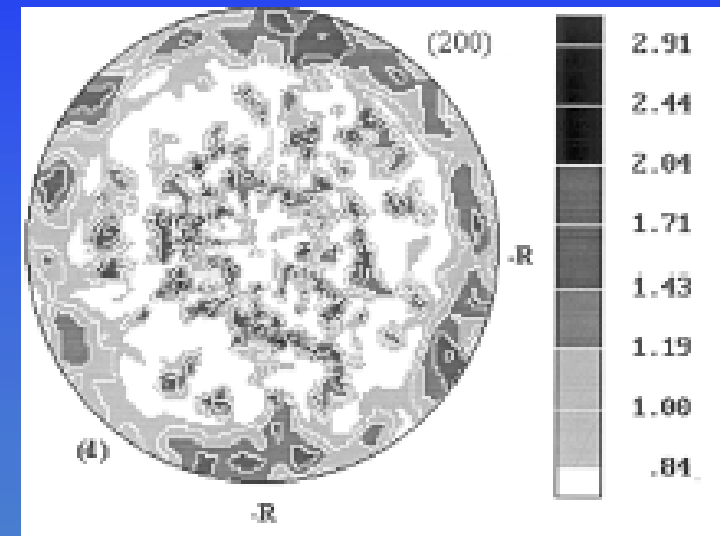


MC Modeling: Application to the Titanium Alloys

Lot A



Lot B

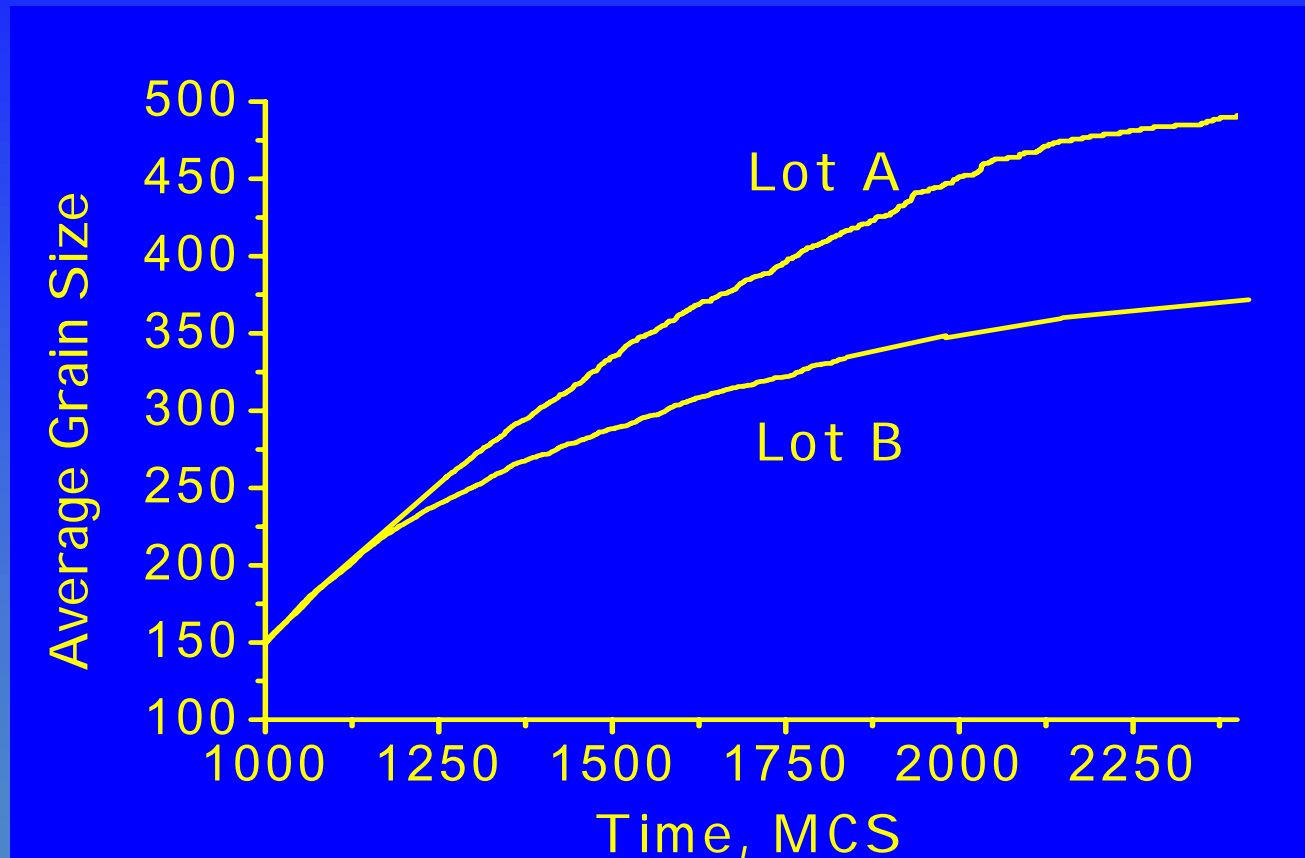


Initial textures¹ of two lots of Ti64 alloy used in simulation

S. L. Semiatin, P. N. Fagin, M. G. Glavicic, I. M. Sukonnik, O. M. Ivasishin
Mater. Sci. Eng. A299, 2001, 225



MC Modeling: Application to the Titanium Alloys



MC simulated grain growth kinetics at isothermal annealing conditions for two lots of Ti64 alloy



Conclusions

- Fine-grained fully β -transformed microstructures processed with Rapid Heat Treatment provide an attractive combination of tensile, fatigue, and fracture characteristics of titanium alloys. Main microstructural features favorably influencing the mechanical properties are shorter length of individual alpha plates and shorter length of individual grain boundary.
- Chemical inhomogeneity of high-temperature beta phase allows gradient microstructures to form which, being optimized, exhibit an improved balance of strength and ductility.



Conclusions (continued)

- Additional cold work has proved to be beneficial in further refining the grain structure using rapid recrystallization annealing. Grain sizes of the order of $10\text{ }\mu\text{m}$ and below required to maximize strength whilst maintaining ductility has been received.
- Modulated on nanoscale level structure of a'' martensite was explained from thermodynamic point of view. It has proved to be responsible for high level of strength properties.



Conclusions (continued)

- Grain growth during beta annealing of titanium is strongly affected by texture. Periods of rapid and slow growth can be related to the evolution of texture during annealing.
- Many features of such texture-controlled grain growth can be reproduced using an advanced Monte-Carlo (MC) modelling technique that was developed to clarify the interaction of grain growth and texture evolution.



Problems Encountered

Technical:

- special equipment adjusted to sizes is needed

Metallurgical:

- results achieved strongly depend on many parameters of little consequence during conventional thermal processing
- continuous phase and structural transformations need “in-depth” understanding of the effect of heating rate



Acknowledgements

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